December 1957 Pages 533-576

The Journal
of the
Society of Dyers and Colourists

1959 LONDON CONGRESS
FIRST VISIT TO ENGLAND
of the
International Federation of
Associations of Textile Chemists
and Colourists
See announcement on page xli

Volume 73 Number 12

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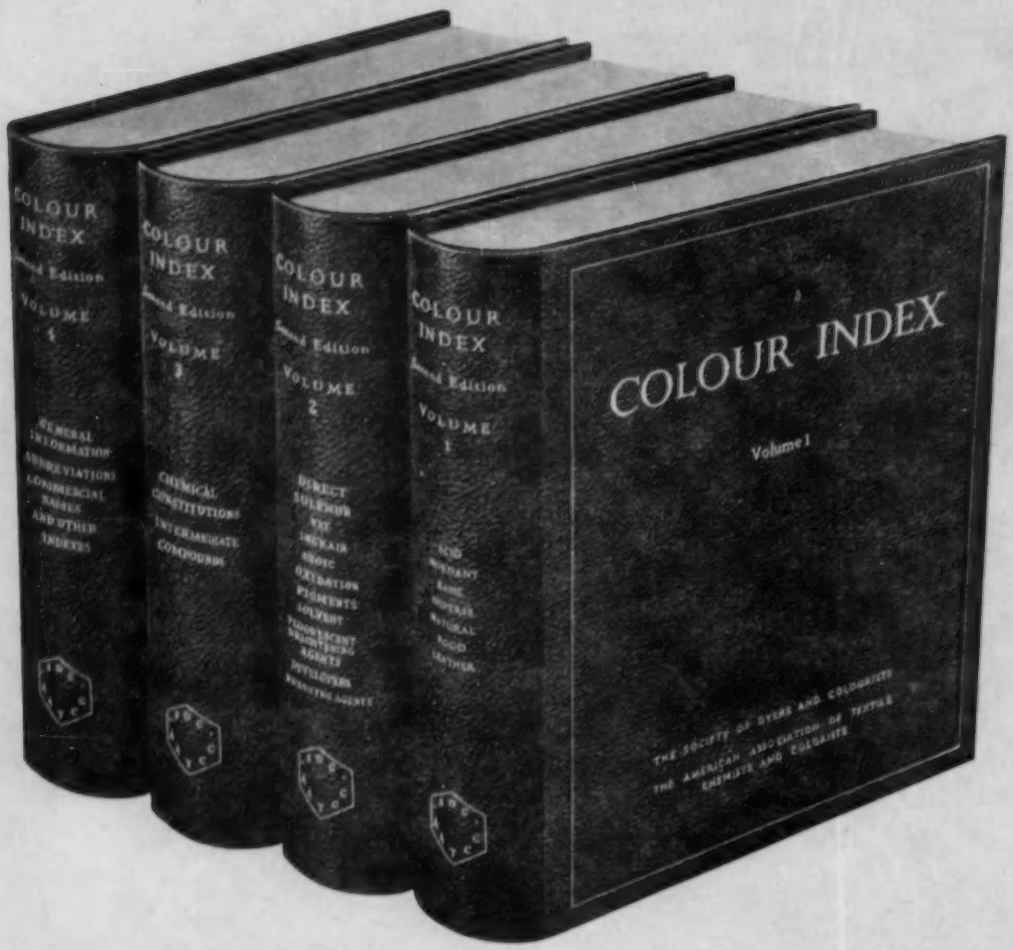

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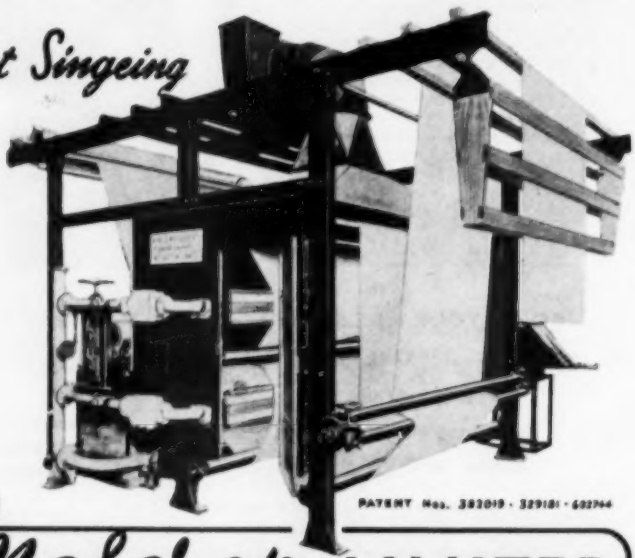

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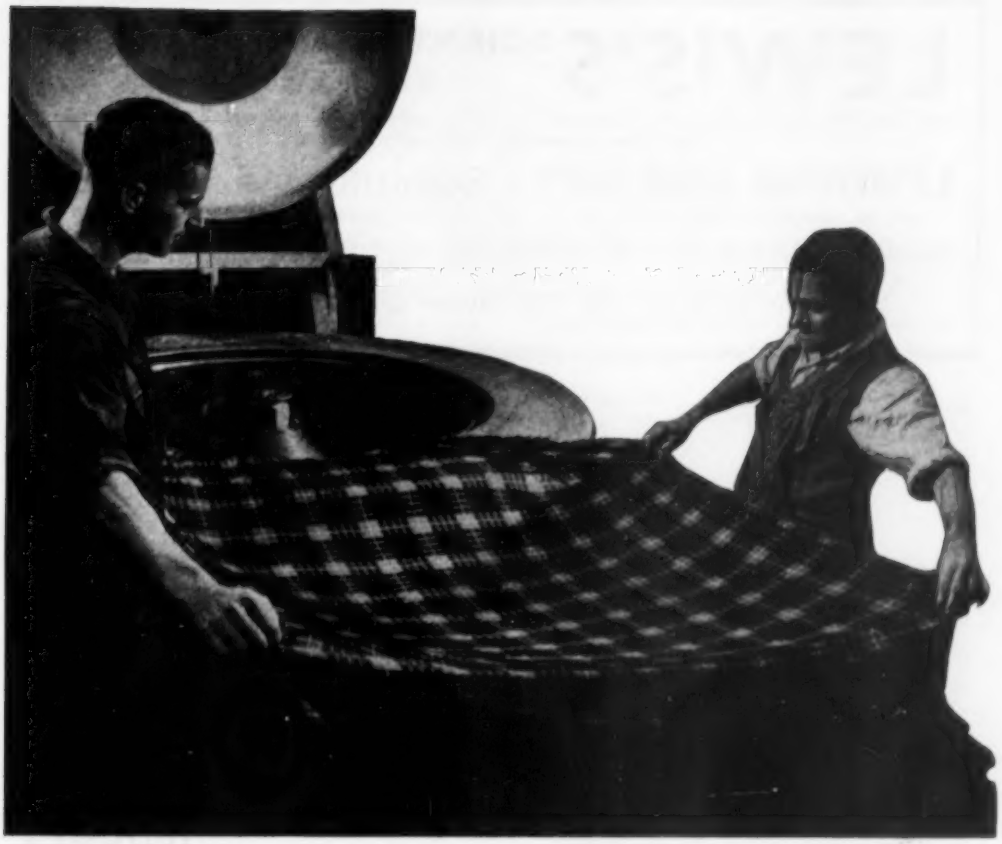

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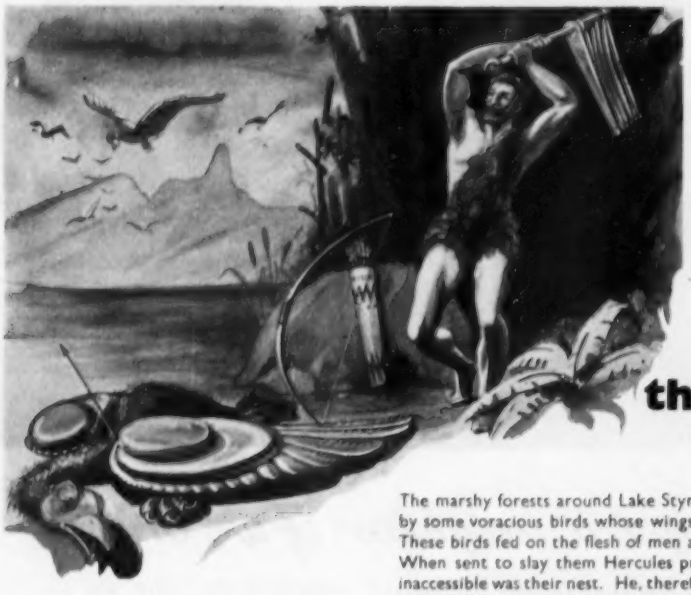

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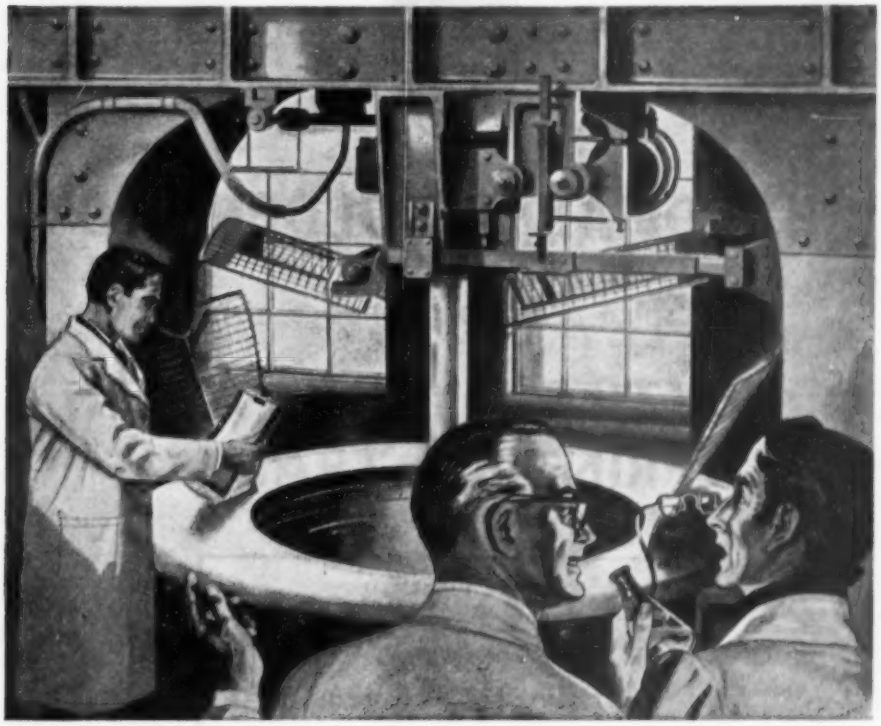

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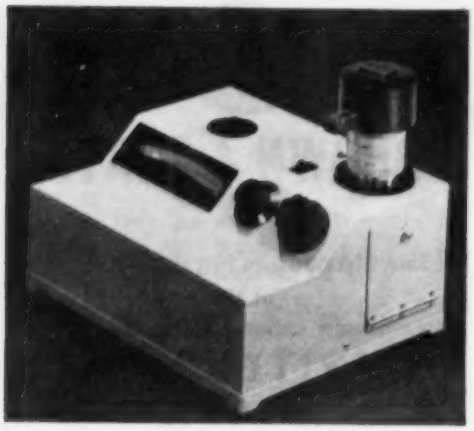

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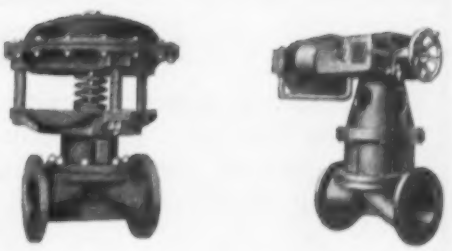

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–7 of the January 1957 and pages 285–290 of the July 1957 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The History and Development of Compressive Shrinkage — *F. V. Davis*

The Application of Pigments to Textiles — *W. Kass*

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JOURNALS WANTED

The Society is urgently wanting Journals for all months of 1955 except July and December. The months of January, February, March, August and September 1955 are particularly required. Copies of January, February, March, April, July, September, October and December 1956 issues are also in demand.

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FORTHCOMING MEETINGS OF THE SOCIETY

Friday, 10th January 1958

LONDON SECTION. *Colour for the Dyer.* Professor W. D. Wright (Imperial College of Science and Technology). (Joint meeting with the London Section of the Oil and Colour Chemists' Association.) Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 14th January 1958

NORTHERN IRELAND SECTION. *The Chemical Foundations of Fast Colour Printing.* A. S. Fern, Esq., B.Sc., D.I.C., F.S.D.C. (Imperial Chemical Industries Ltd.). Further details later.

SCOTTISH SECTION. *The Application of Fluid Beds to Textile Processing.* (Joint Meeting with Textile Institute.) J. Wilson, Esq., M.C., M.Sc., F.R.I.C. (British Rayon Research Association). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Thursday, 16th January 1958

MANCHESTER JUNIOR BRANCH. *High Temperature Dyeing on Synthetic Fibres.* H. R. Hadfield, Esq., M.Sc.Tech. Room C9, Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. *Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Victoria Hotel, **Bradford.** 7.30 p.m.

Friday, 17th January 1958

MANCHESTER SECTION. *The Chemical Foundations of Fast Colour Printing.* A. S. Fern, Esq., B.Sc., D.I.C., F.S.D.C. (Imperial Chemical Industries Ltd., Dyestuffs Division). The Textile Institute, 10 Blackfriars Street, **Manchester 3.** 7 p.m.

LONDON SECTION. Dinner and Dance. Waldorf Hotel, **London, W.C.2.**

Tuesday, 21st January 1958

HUDDERSFIELD SECTION. *Tests for Colour Vision.* F Jordinson, Esq., M.Sc., F.R.I.C., F.S.D.C. and T. Minshall, Esq., A.M.C.T., A.T.I. (Dyeing Department, Technical College, Huddersfield). (Joint Meeting with the Huddersfield Section, Royal Institute of Chemistry.) Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

SCOTTISH SECTION. *Flame Resistant Finishes.* J. R. W. Perfect, Esq., B.Sc. (Bradford Dyers' Association Ltd.). Developments with the use of phosphorus-containing resins. St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 22nd January 1958

MIDLANDS SECTION. *Colloquium . . . Auxiliary products—Use and Abuse.* (Short papers by local members.) College of Technology, **Leicester.** 7 p.m.

SCOTTISH JUNIOR BRANCH. *Practical Aspects of Fastness Testing To-day.* P. R. Dawson, Esq. (Imperial Chemical Industries Ltd.). Technical College, **Paisley.** 7.30 p.m.

Friday, 24th January 1958

WEST RIDING SECTION. Ladies' Evening. Victoria Hotel, **Bradford.** 7.30 p.m.

Monday, 27th January 1958

BRADFORD JUNIOR BRANCH. *Spun-dyed Fibres, their Properties and Uses.* C. C. Wilcock, Esq., A.R.T.C., F.T.I., F.S.D.C. (Courtaulds Ltd.). Bradford Institute of Technology, **Bradford.** 7.15 p.m.

Tuesday, 28th January 1958

LEEDS JUNIOR BRANCH. *Organic Pigments.* F. M. Smith, Esq., Ph.D. Department of Colour Chemistry and Dyeing, The University, **Leeds 2.** 3.15 p.m.

Thursday, 30th January 1958

WEST RIDING SECTION. *A New Approach to Pigment Dyeing of Piece Goods.* T. Weber, Esq. (Ciba Ltd., Basle). Victoria Hotel, **Bradford.** 7.30 p.m.

Saturday, 1st February 1958

MANCHESTER SECTION. Annual Dinner and Dance. Grand Hotel, **Manchester.** 6 p.m. for 6.30 p.m.

Monday, 3rd February 1958

HUDDERSFIELD SECTION. *Shrink Resistance of Wool.* A. N. Davidson, Esq., B.Sc. (Wool Industries Research Association). (Joint Lecture with the Huddersfield Textile Society.) Technical College, **Huddersfield.** 7.30 p.m.

Tuesday, 4th February 1958

LONDON SECTION. *Progress in Textile. A review of developments and their effect on industry and user—Dyeing and Finishing.* T. Vickerstaff, Esq., M.Sc., Ph.D., F.S.D.C. (Joint Meeting with the London Section, Textile Institute.) Chemical Society, Burlington House, **London, W.1.** 6.30 p.m.

Friday, 7th February 1958

LONDON SECTION. *Spun-dyed Fibres, their Properties and Uses.* C. C. Wilcock, Esq., A.R.T.C.S., F.T.I., F.S.D.C. (Courtaulds Ltd.). Royal Society, Burlington House, **London, W.1.** 6 p.m.

Monday, 10th February 1958

NORTHERN IRELAND SECTION. *A New Approach to Pigment Dyeing.* T. Weber, Esq. (Ciba Limited). Further details later.

Tuesday, 11th February 1958

BRADFORD JUNIOR BRANCH. *The Role of Optical Whitening Agents.* C. Fearnley, Esq., B.Sc., Ph.D. (Geigy Co. Ltd.). Bradford Institute of Technology, **Bradford.** 7.15 p.m.

SCOTTISH SECTION. *A New Approach to Pigment Dyeing of Piece Goods.* T. Weber, Esq. (Ciba Limited). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Thursday, 13th February 1958

MANCHESTER JUNIOR BRANCH. *The Scientific Background to Mangling.* E. Moss, Esq. Room C9, Manchester College of Technology, **Manchester.** 4.30 p.m.

MIDLANDS SECTION. *A New Approach to the Pigment Dyeing of Piece Goods.* T. Weber, Esq. (Ciba Ltd, Basle.) Gas Board Theatre, **Nottingham.** 7 p.m.

WEST RIDING SECTION. *Investigations on the Dyeing Process of Polyester Fibres with Disperse Dyes.* Dr. Glenz (Bayer Co., Leverkusen). Metropole Hotel, King Street, **Leeds.** 7.30 p.m.

Tuesday, 18th February 1958

HUDDERSFIELD SECTION. *The Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

Wednesday, 19th February 1958

SCOTTISH JUNIOR BRANCH. Lecture by a representative of The British Launderers' Research Association. (Title to be announced later.) (Joint meeting at the invitation of The Textile Institute.) Technical College, **Paisley.** 7.30 p.m.

Thursday, 20th February 1958

BRADFORD JUNIOR BRANCH. Annual Dance. Fountains Hall, **Bradford.**

Friday, 21st February 1958

MANCHESTER SECTION. *The Application of Acrylonitrile in the Textile Industry.* J. H. MacGregor, Esq., Ph.D., F.R.I.C. (Courtaulds Ltd., Textile Research Laboratory, Bocking). The Textile Institute, 10 Blackfriars Street, **Manchester 3.** 7 p.m.

Tuesday, 25th February 1958

LEEDS JUNIOR BRANCH. Title to be announced later. Miss E. Smith., The Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds 2.** 3.15 p.m.

Thursday, 27th February 1958

WEST RIDING SECTION. J. B. Speakman, Esq., D.Sc., F.R.I.C., F.T.I. Details later.

continued on page xxxvii

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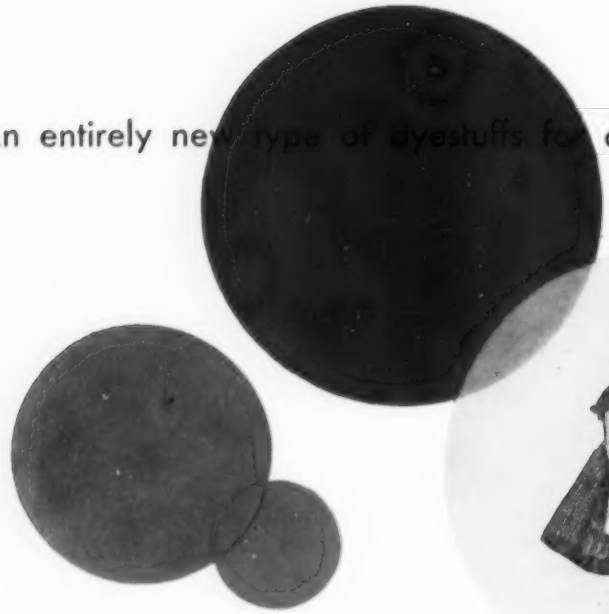

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THE JOURNAL
OF THE
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Volume 73 Number 12 DECEMBER 1957 *Issued Monthly*

Proceedings of the Society

Applications of Colour Physics to Textiles

J. C. Guthrie and P. H. Oliver

Meetings of the Midlands Section held at the College of Technology, Leicester, on 22nd November 1956, Mr. C. A. Mills in the chair; of the Northern Ireland Section held at Thompson's Restaurant, Belfast, on 9th January 1957, Mr. R. J. G. Reid in the chair; of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 15th January 1957, Mr. W. G. B. Grant in the chair; of the Huddersfield Section held at Silvio's Café, Huddersfield, on 22nd January 1957, Mr. J. Calvert in the chair; and of the West Riding Section held at the Hotel Metropole, Leeds, on 14th February 1957, Mr. C. W. Green in the chair

Both additive and subtractive mixing of colours are discussed, leading to the C.I.E. system of colour specification, which is based on three primary colours and additive mixing. Measuring instruments and some types of illuminants are considered. Applications include examples of metameric matches, dichroism, quality control, and some problems in the blending of coloured fibres.

In the November 1953 issue of the *Journal* of this Society details were given of regulations governing the election of Associates[1]. Under the heading *Examination* is Paper B, the scope of which includes theories of colour, relation of colour to constitution, colour assessment, and colour fastness. Examination papers which have been set since 1953 give a clearer idea of the scope of this paper[2]. The established dyer may, however, be wondering why it has become necessary for colour physics to be studied and is probably doubtful of its value. This paper is designed to give an overall survey of the field and will necessarily deal with various topics in a very superficial way.

A great deal of thought and labour has been devoted to attempts to establish a precise terminology for colour, so as to avoid confusion during discussions. The Physical Society published in 1948 a *Report on Colour Terminology*[3]. After defining terms used in colour physics, colour vision, etc., the Report gives definitions of terms used in various industries including that of dyeing. Members of the Society of Dyers and Colourists were included on the committee. International agreement on colour terms and definitions is being attempted by the Commission Internationale de l'Éclairage (C.I.E.).

Many important terms that can easily be defined in relation to technical processes have no exact counterpart in colour physics. The word *colour* relates both to the objective qualities or physical properties of the stimulus and to the subjective sensation arising in the eye or the brain. In the Second John Mercer lecture, White and Vickerstaff[4] discussed *Colour* in some detail.

The most important terms relating to colour quality which will be used in this paper are *hue*, *saturation*, and *lightness*. *Hue* is the quality which differentiates the colours in the spectrum and is related to wavelength, e.g. red, green, blue, etc. *Saturation* refers to the quality which differentiates the colours by the amount of grey which can be regarded as present in them, a spectral colour having the highest and a grey zero saturation. *Lightness* can be understood by reference to the variation from black to white through the range of greys that fall between them; this concept can be applied also to colours other than greys.

Basic Principles of Colour Specification

Fig. 1 shows the position of the visible spectrum on the wavelength scale of electromagnetic waves. The wavelengths of visible light extend between about 4×10^{-5} cm. or 400 mμ.* for the extreme violet and $7{\cdot}2 \times 10^{-5}$ cm. or 720 mμ. for the far red. The eye is not sensitive to radiation of wavelength less than 400 mμ. or greater than 720 mμ. The visible spectrum which may be obtained by passing white light through a prism covers only a very small part of the whole range of electromagnetic waves.

Additive and Subtractive Mixing

Fig. 2 illustrates the two different methods of mixing colour. Additive mixture occurs when two or more beams of coloured light are superimposed on a screen, or when two or more sectors of a colour top fuse visually as the top rotates.

* Wavelengths of light are usually expressed either in Ångström units or "tenth-metres" (1 Å. = 10^{-10} m. = 10^{-8} cm.) or in millimicrons (1 mμ. = 10^{-9} m. = 10^{-7} cm.).

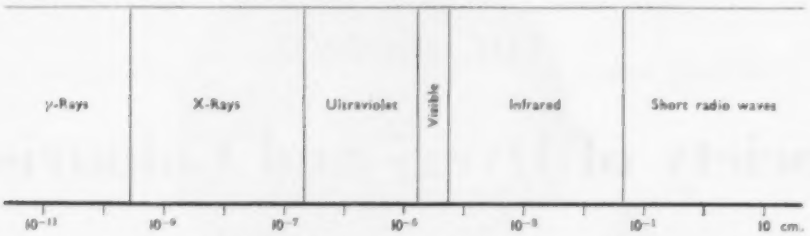


FIG. 1—Scale of Wavelengths for the Range of Electromagnetic Waves

Suitably chosen primary colours will produce white light by additive mixture since they are adding together different portions of the spectrum. Subtractive mixture occurs when transparent coloured layers are superimposed. Then light transmitted by the combination has subtracted from it those portions of the spectrum absorbed by each layer. Subtractive mixture, produced e.g. by superimposing continuous layers of transparent coloured inks on white paper, which reflects light through the layers, may produce black if the combination is capable of absorbing strongly every portion of the spectrum.

The primary colours for additive mixing are red, green, and blue (R, G, and B). These can be made to yield a large number of colours by altering the intensities of the three components. In dyeing, the three primary colours are magenta, yellow, and cyan, and subtractive mixing occurs.

TRICHROMATIC SPECIFICATION

The quantitative system of colour measurement is based on additive mixing of red, green, and blue, and is known as the *C.I.E. system* [5]. There is no system of quantitative specification possible which would combine both simplicity and generality based on subtractive mixing. The C.I.E. system came into effect in 1931 and can be applied to dyeing problems, although the latter mixings are subtractive. It follows that complementary colours may be of two types, giving achromatic sensations by additive or subtractive mixing respectively.

The additive spectral primaries commonly used are red 700·0 mμ., green 546·1 mμ., and blue 435·8 mμ. Other colours, corresponding to mixtures of any two, will fall on the line connecting the two primaries. Thus the line joining red and green in Fig. 3 is the locus of all colours obtained by mixing these two primaries. Colours which require all three primaries for matching fall inside the colour triangle. Conversely, each point on the triangle represents a colour obtained by mixing definite proportions of R, G, and B. The white or grey point is obtained from equal proportions of each of the three primaries.

A difficulty arises with spectral colours, since these fall outside the triangle, and it is advantageous to deal with components X, Y, Z instead of R, G, B. It is convenient to regard X as a red more highly saturated than spectrum red, Y more saturated than spectrum green, and Z more saturated than spectrum blue. Alternatively, X, Y, Z may be regarded as imaginary primaries.

The result of the mathematical transformation is that the unknown colour is specified in terms of the new reference primaries X, Y, Z. Chromaticity coordinates are obtained as follows—

$$x = \frac{X}{X+Y+Z} \quad y = \frac{Y}{X+Y+Z} \quad z = \frac{Z}{X+Y+Z}$$

x can be plotted against y as in Fig. 4, and z is known since $x + y + z = 1$.

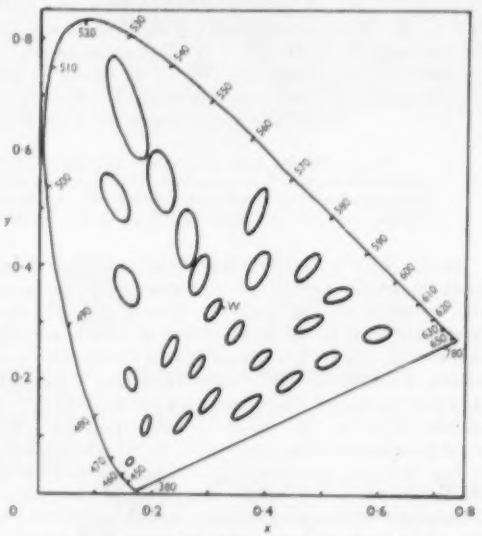


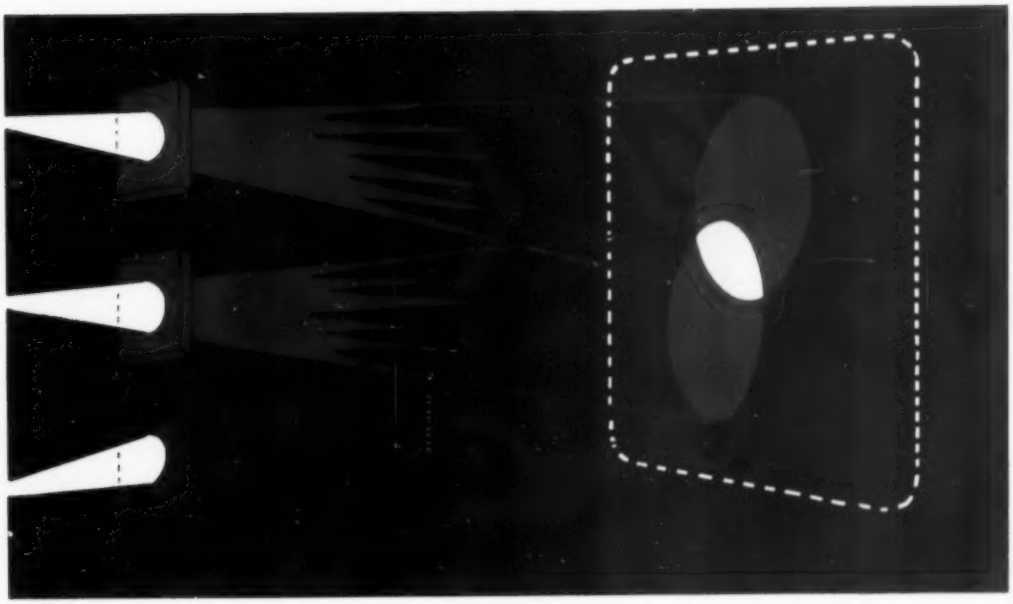
FIG. 4—Chromaticity Diagram and Spectrum Locus (wavelengths in mμ.)

Chromaticity coordinates can be regarded as the fractional amounts of the three primaries required to match a given colour. In Fig. 4, movement in the x direction corresponds to an increase or a decrease in the proportion of red, and movement in the y direction corresponds to variation in green. The white point W is given by—

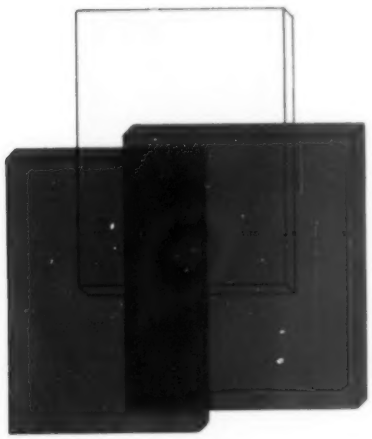
$$x = \tfrac{1}{3} \quad y = \tfrac{1}{3} \quad z = \tfrac{1}{3}$$

The continuous curve is the locus of spectrum colours from blue through green to red. It is usual to draw a straight line across the ends of the curve, and colours in the region of this line are purples. In setting up the C.I.E. system it was arranged mathematically for Y to be a measure of lightness as well as greenness, and colour is quantitatively specified by x, y, and Y.

These three quantities x, y, Y can now be plotted along three axes and a colour solid obtained. In practice the solid may be plotted on other axes, the coordinates being calculated from x, y, Y, in



Additive Colour Mixture



Subtractive Colour Mixture

FIG. 2.—Additive and subtractive Colour Mixture

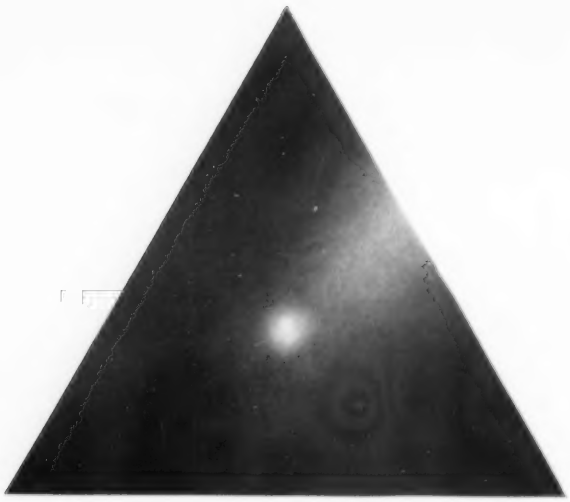


FIG. 3 — Colour Triangle (the central spot where the three colours merge and become white has been deliberately emphasised, in order to illustrate more clearly the fact that perfect mixing of three primary colours can produce white)

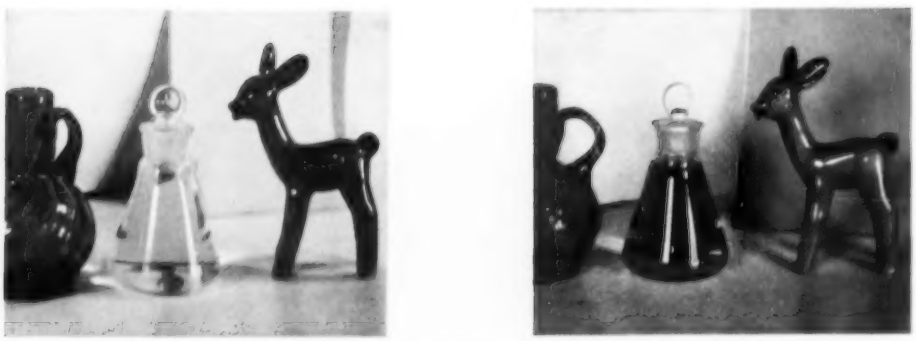


FIG. 7 — Effect of Spectral Energy Distribution of Illuminant on Colour of Objects

(1) (3)

FIG. 11 — Colour of Different Thicknesses (1 and 3) of Dyed Cellulose Acetate Film

FIG. 13 — Production of Complementary Afterimage

order to give a visually more uniform colour distribution. Fig. 5 gives an idea of the arrangement of some colours in a colour solid, with black at the base, white at the apex, and greys along the line joining black and white.

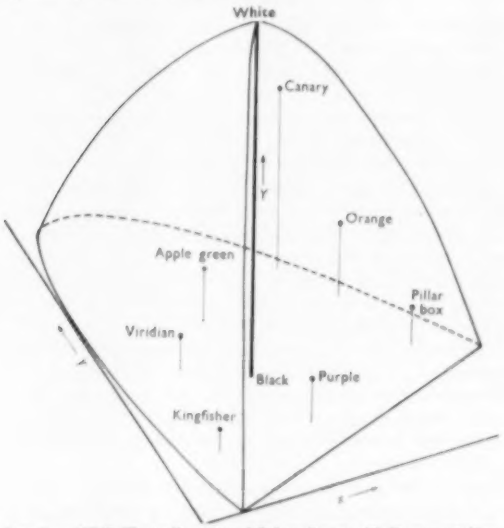


FIG. 5A— C.I.E. Three-dimensional Colour Space with Representative Colours plotted (*Y* is the dimension perpendicular to the *x*–*y* plane)

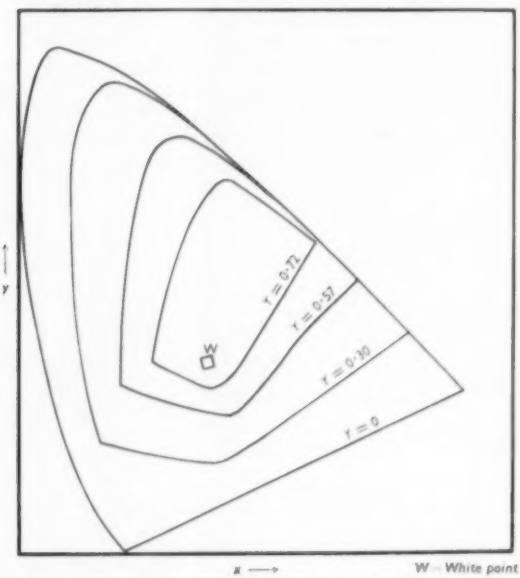


FIG. 5B— Horizontal Chromaticity Planes at Various Values of Lightness *Y*

By plotting representative dyes and pigments in the colour solid, it has been found that reds, oranges, and yellows approach most closely the theoretical maximum saturation, which can be calculated. It can be shown that this is due to their spectral absorption curves approximating to the ideal shape, which is rectangular. Greens and blues fall short in this respect. Fuller details on colour gamuts for various types of dyes are available [6].

INSTRUMENTS

Commercial instruments for colour measurement fall into two classes— (1) spectrophotometers [7, 8] and (2) colorimeters [9–13].

When a coloured surface is investigated with a spectrophotometer the reflectance curve is usually drawn. This is obtained by illuminating the surface with a narrow waveband from the spectrum and comparing the amount of light reflected from the surface with the amount reflected from a white surface. The white (usually magnesium carbonate) reflects approx. 100% of light at all wavelengths. By working across the spectrum from violet to far red in a series of narrow wavebands, which are usually 1–10 mμ. wide, a series of points is obtained which define the reflectance curve for the surface. Some instruments are fitted with pen recorders, and the reflectance (or transmission) curve is drawn automatically.

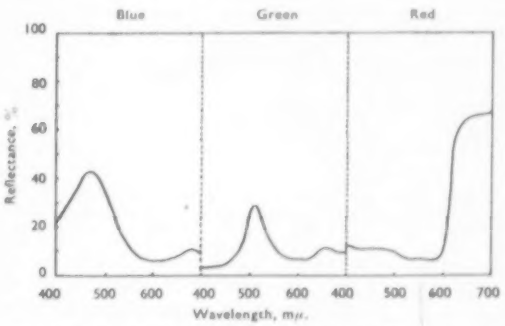


FIG. 6— Typical Reflectance Curves for Pigmented Viscose Rayon

Some typical reflectance curves are shown in Fig. 6. A pure white surface reflects 100% at all wavelengths; a black surface reflects 0%; and a neutral grey has a constant reflectance below 100% at all wavelengths.

The reflectance or transmission curve gives the maximum amount of information on the colour. From this curve the values of *X*, *Y*, *Z* may be calculated [14], or alternatively some spectrophotometers are fitted with special devices which automatically evaluate *X*, *Y*, *Z* while the curve is being drawn. On the other hand, recording instruments are expensive, costing several thousands of pounds, and may well be difficult to justify for the small dyehouse. Non-recording spectrophotometers are much less expensive than recording types. It should be noted that the accuracy of these instruments is not all that could be desired for surfaces of low reflectance.

The alternative to the spectrophotometer is a tristimulus colorimeter, which may sacrifice some accuracy in absolute C.I.E. units, but nevertheless may be at least as accurate from the point of view of reproducibility and the measurement of small colour differences. Values of *X*, *Y*, and *Z* may be obtained by direct measurement.

Recent views on commercial instruments have been summarised by Wright [15].

ILLUMINANTS [4, 16, 17]

The colour seen when looking at an object or when looking through a coloured liquid depends on the spectral quality of the illuminant or light source, and this is defined by the spectral curve of the illuminant, which can be daylight, tungsten light, fluorescent lighting, etc. Sodium lighting, as in street lighting, gives notoriously poor colour effects, since all the radiation is concentrated in bands in the yellow region of the spectrum.

In deciding upon an illuminant for a particular purpose it is essential to know the spectral energy distribution. Fig. 7 shows the same scene illuminated by two light sources. In the upper photograph the illuminant has a continuous spectrum, while in the lower the radiation is concentrated in bands in the red and blue–green parts of the spectrum. Visually these two illuminants were a match [18].

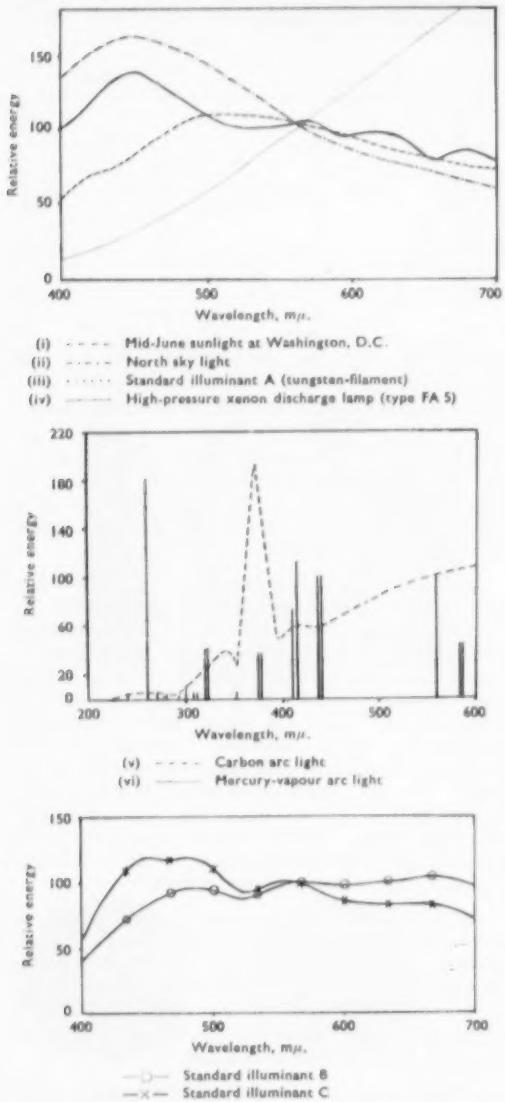

Relative energy
Wavelength, mμ.
(i) Mid-June sunlight at Washington, D.C.
(ii) North sky light
(iii) Standard illuminant A (tungsten-filament)
(iv) High-pressure xenon discharge lamp (type FA 5)
(v) Carbon arc light
(vi) Mercury-vapour arc light
Standard illuminant B
Standard illuminant C

FIG. 8—Spectral Energy Distributions of Various Illuminants [22]

Miscellaneous Applications

LIGHT SOURCES FOR FADING AND PHOTODEGRADATION [16]

Spectral distributions for four illuminants are shown in Fig. 8 and differences in colour-rendering properties between these are quite striking. Daylight itself is of variable quality [19]. The low-pressure mercury arc radiates in certain wavebands only (chiefly 404·7, 407·8, 435·8, 546·1, 577·0, and 579·0 mμ.), and this type of illuminant is not to be recommended for fading or photodegradation experiments in which it is hoped to simulate the effect of daylight; it is also unsuitable as an illuminant for colour matching.

Compared with North sky light, tungsten light is rich in red and deficient in blue. It can be corrected by filters to give standard light sources known as *Illuminant B* (approximately sunlight) and *Illuminant C* (approximately North sky light) [20]. The necessary filters cut down the intensity by such a large factor that use of these two latter illuminants is not practicable for fading or photodegradation work.

The carbon arc is used in fading lamps [21] and also for photodegradation. It has an undesirable peak in the near ultraviolet, which may give rise to errors in light-fastness assessment in some cases.

The xenon arc has a spectral energy distribution which approximates to daylight [22] and should be suitable for fading and photodegradation work. Few experimental data on its use with textiles are available, although one commercial fading lamp employing a xenon arc as illuminant is available.

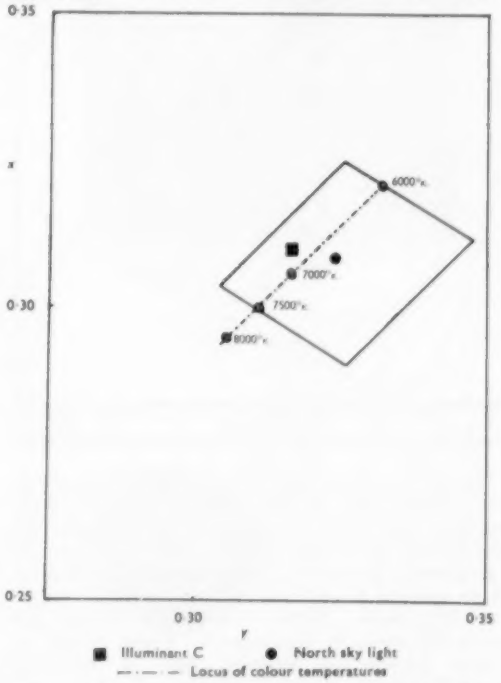


FIG. 9—Chromaticity Specification of Colour-matching Lamps [23]

SPECIFICATION OF COLOUR-MATCHING LAMPS [23]

The usual standard lighting for colour matching is North sky light. It has been shown how important the colour of the light is in its effect on the colour of samples. It is thus desirable to fix standards for the illuminant. North daylight can be variable in quality depending on cloud conditions, time of day, and proximity of coloured walls facing the outside of the window.

Fluorescent colour-matching lamps are designed, by using suitable phosphors, to correspond in colour to average North sky light. It is important that definite specifications are available which will ensure that the colour-rendering properties lie within close limits. The specification is laid down firstly in terms of the chromaticity diagram (Fig. 9). This specification alone is not sufficient, since quite different spectral distributions can give the same chromaticity. The colour rendering, on the other hand, depends on the spectral distribution, and for this reason it is necessary to specify limits for the energy in each of eight wavebands [24].

METAMERIC MATCHES

If two patterns have the same values of x, y, and Y for a given illuminant, i.e. if they match, they will not necessarily match in another illuminant unless their reflectance curves are exactly alike. Two patterns having quite different reflectance curves may match under one illuminant but will not be a match when viewed under other illuminants of different spectral characteristics. When this occurs, the match is said to be *metameric*.

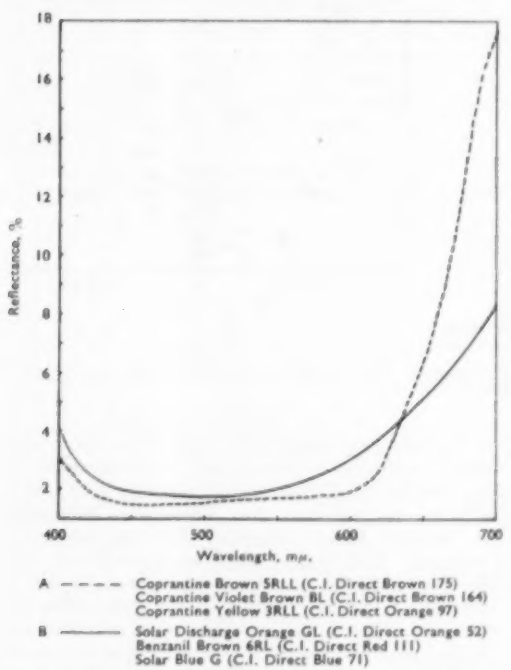

Reflectance, %
Wavelength, mμ
A ---- Coprantine Brown 5RLL (C.I. Direct Brown 175)
Coprantine Violet Brown BL (C.I. Direct Brown 164)
Coprantine Yellow 3RLL (C.I. Direct Orange 97)
B ——— Solar Discharge Orange GL (C.I. Direct Orange 52)
Benzanil Brown 6RL (C.I. Direct Red 111)
Solar Blue G (C.I. Direct Blue 71)

FIG. 10—Two Dye Mixtures showing Reversal of Colour under Different Illuminants

An interesting case is illustrated in Fig. 10. In tungsten light, which is rich in red and far red, A is redder than B, owing to its higher reflectances around 700 mμ. With a colour-matching fluorescent lamp which is deficient at the far red end of the spectrum, the pattern B is the redder. This is because the bulk of the red radiation in this fluorescent lamp is in the region below about 630 mμ., and so the effective parts of the curves, this time, are wavelengths less than this, where curve B has higher reflectance values than A.

COLOUR MATCHING

Several papers have been published describing attempts to predict recipes for dyeing. We have only a limited experience ourselves of these methods, but it is possible to state that the final match cannot at present be predicted with sufficient accuracy. It is possible, however, to obtain an approximate match, which can then be corrected by the dyer. The small amount of work reported has usually referred to the dyeing of acid dyes on wool. It is important to realise that colorimetric methods usually give the amount of dye on the fibre, and not the concentration of dye required in the dyebath. In this country some work has been carried out by Atherton [25], who refers to attempts at colour matching by other authors.

The present position is that the replacement of the dyer by instruments for obtaining recipes is not likely to occur for some time. On the other hand, aid can be given, chiefly in cutting down a large number of trial dyeings, by plotting the results of these as he proceeds.

DICHROISM

Dichroic colour changes are sometimes met when dyes are used in varying concentrations on a given thickness of substrate, or when the thickness varies

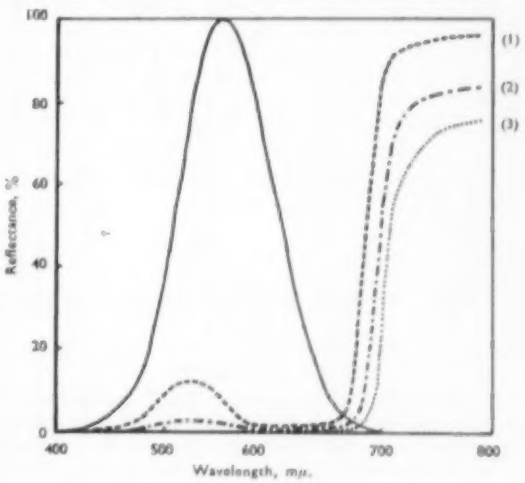

Reflectance, %
Wavelength, mμ
(1)
(2)
(3)
——— Sensitivity curve of the normal human eye (relative lightness, %)
(1) ---- (2) ---- (3) ..... Reflectance curves of 1, 2, and 3 layers of dyed cellulose acetate film

FIG. 12—Reflectance Curves showing Effect of Concentration of Dye or Thickness of Film on the Colour of Dyed Cellulose Acetate (see Fig. 11)

with the dye concentration constant. If a sheet of secondary cellulose acetate is dyed with Dispersol Fast Yellow G (C.I. Disperse Yellow 3) and Duranol Brilliant Blue BN (C.I. Disperse Blue 3) in the ratio of approx. 2 : 1, the sheet may be dyed green. If several thicknesses of this sheet are superimposed on each other and a tungsten lamp is viewed through the combination, red is seen (Fig. 11).

The transmission curve for one thickness of sheet shows a peak in the green region (Fig. 12), and this, combined with the higher sensitivity of the eye in this region, swamps the red, although the latter has greater transmission. This is due to the lower sensitivity of the eye for red, as shown by the visibility curve. When three thicknesses of the sheet are used, the transmission of the sheet in the green disappears completely, and the only light transmitted is at the red end of the spectrum. Hence a low-intensity red light is seen.

industry. For the simplest case, the coloration of a fibre with a single dye, we have constructed an instrument for sorting pirns of spun-dyed (Duracol) continuous-filament viscose rayon. This type of measurement requires previous calibration to set up limits corresponding to just perceptible differences, and this may be a disadvantage if only short runs of a given colour are to be checked.

Again, if one standard colour is measured and the chromaticity is plotted on the diagram, then the ellipses shown in Fig. 4 represent the relative sizes of the regions in which colours must fall in order that they are not perceptibly different. In Fig. 4 the ellipses are ten times as large as the actual discriminations[27]. It is necessary to combine the data plotted on the diagram with data on any lightness variations. An investigation on colour discrimination has also been published by Wright[28] and a summary has been given by Horner[29].

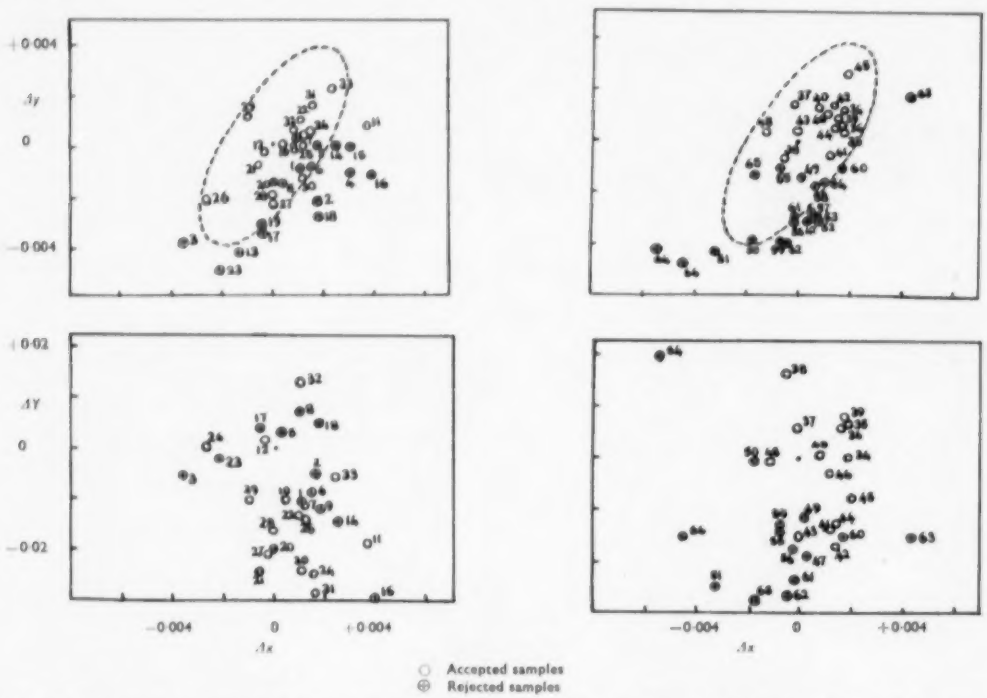


FIG. 13—Colorimetric Specifications of Samples in Illuminant C—two diagrams were plotted in each case (Δy and ΔY) merely to avoid overcrowding[30]

ABSORPTIOMETRY

Absorptiometry is a field in which applications of colorimetry have proved of value. For many years, optical measurements on dye solutions have been in use for checking dyebath exhaustions, etc. We do not propose to discuss the many applications of these techniques, since they have been covered in various lectures and papers from time to time[26].

QUALITY CONTROL

Quantitative specifications of colour have not been used to any great extent in the textile

The following example is taken from the acceptance or rejection of 66 samples of Silvertan Shade No. 193 by the U.S.A.A.F.[30] on grounds of colour only, i.e. not fastness, etc. These were submitted by various firms competing for the particular contract. The dotted lines in Fig. 13 are the calculated MacAdam ellipses representing a certain number of just perceptible colour steps from the standard. The samples which were accepted are plotted as open circles; the ones plotted as crosses in circles were rejected. These dotted ellipses are in good agreement with the U.S.A.A.F. colour

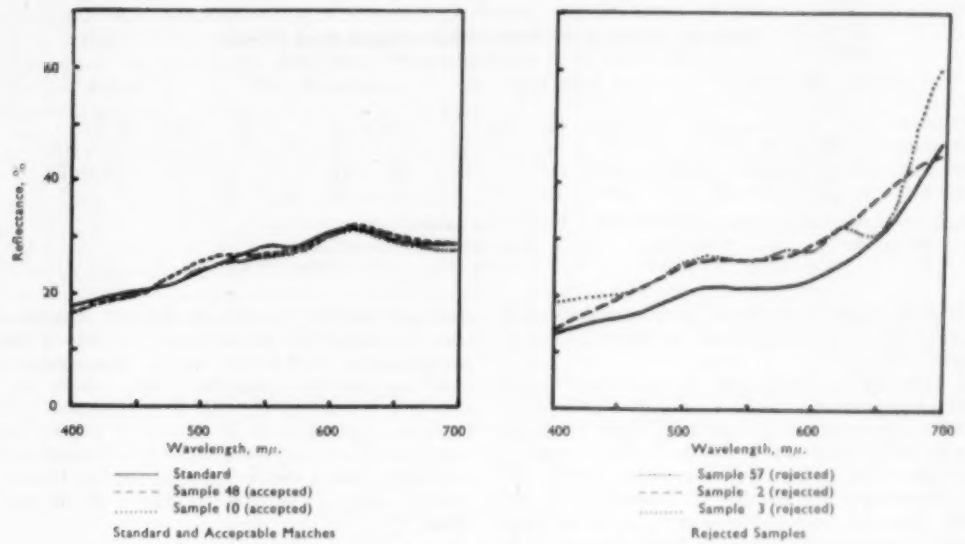


FIG. 14—Spectrophotometric Curves of Standard and Samples[20]

passers. The lower diagrams show that no samples were rejected because of the ΔY values: i.e. the colour passers were concerned mainly with chromaticity differences, and accepted large variations in lightness of the samples submitted.

In order to pass for all types of illuminant, the reflectance curves of the patterns should be near that of the standard: i.e. the choice of dyes is important (Fig. 14).

It was concluded in this case that, if the spectrophotometric curve was nearly identical with that of the standard, the pattern would be accepted as a colour match. Also, tolerances could be quoted on the chromaticity data, but these alone would not ensure acceptance. The incorrect choice of dyes was apparently the most important cause of rejection in this case.

In the more general case where checking is to be carried out on dyeings obtained with several dyes, it is possible to use trichromatic measurements for tolerance control. Any set of three filters—red, green, and blue—should serve the purpose when the colour differences are small, though it is preferable to standardise on a universal trichromatic system, which is the advantage of the C.I.E. system.

Blends of Coloured Fibres

Coloured fibres may be blended at various stages of the spinning process in order to produce a variety of effects. For a 50:50 mixture of yellow and blue Fibro-Duracol, effects ranging from yellow warp and blue weft yarns to an intimate mixing of the yellow and blue fibres, which gives an overall green effect, can be obtained. For most intimate mixing, on the cotton system, blending should take place at the card. Drawframe blending gives a less intimate mixing, and reflectance curves for blending

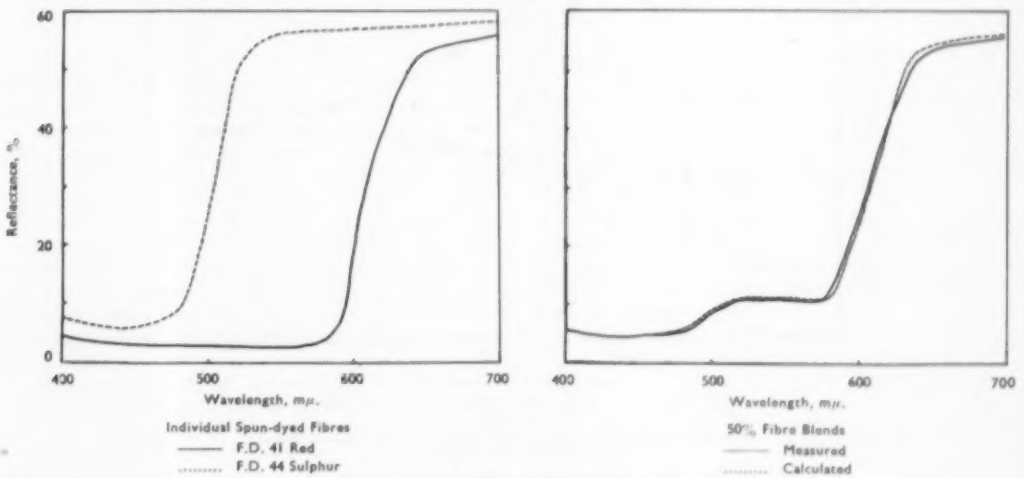


FIG. 15—Blending of Spun-dyed Fibres

TABLE I*

Colours of Blends of Fibro-Duracol Spun-dyed Fibres†

(The values for X and Z refer to Y = 0·10–0·15)

X ... / Z	0·08–0·09	0·09–0·10	0·10–0·11	0·11–0·12
0·03–0·06	—	1B + 2Y + 1G	3B + 7Y	1B + 3Y
0·06–0·09	3G + 1S	1Y + 1G + 2S	1Y + 2G + 1S	2Y + 1G + 1LB
0·09–0·12	3G + 1LB	3G + 1T	7G + 3T	2Y + 1LB + 1T
0·12–0·15	1G + 1T + 2S	2R + 1G + 3T	1G + 2S + 1F	2G + 1S + 1F

* Sample extract from colour catalogue compiled by Dr. J. Crank (private communication).

† B = Medium blue; F = Bright Fibro; G = Apple green; LB = Light blue; R = Red; S = Sulphur yellow; T = Turquoise; Y = Indian yellow

at various stages have been given by Warburton and Lund[31]. The card blend is nearest to subtractive mixing.

If blending is carried out at the card, so that subtractive mixing is approached, it is possible to calculate the colour of the resulting blend by means of a formula suggested by Friele[32]. Fig. 15 shows reflectance curves for red and yellow fibres and also curves for the mixture (i) calculated by the Friele formula, and (ii) measured on a Uvispek (Hilger) spectrophotometer. This agreement is quite good. It is possible to calculate the resultant colour of a three-component blend once the reflectance curve for each of the components is known.

COLOUR CATALOGUE BY CALCULATION

The reverse process of calculating the amounts of three components for a blend to match a pattern is at present impracticable. This difficulty may be side-tracked provided that a large amount of preliminary calculation is carried out. The resultant colour of a blend of three components may be calculated for a large number of blend compositions, and these can be catalogued. In order to use the catalogue, the pattern to be matched is measured with a tristimulus colorimeter to determine its X, Y, and Z values; these values are looked up in the appropriate place in the catalogue, and a recipe is found for the blend. A sample page of such a catalogue is shown in Table I*.

The production of the catalogue for blends of, say, eighteen Fibro-Duracol colours is a fairly large task, but it is very much easier than the corresponding task of producing an adequate catalogue with the samples mounted in it for visual matching. It must be emphasised that the catalogue is compiled mathematically, so that no actual mixing of fibres need take place for its preparation. This type of approach could possibly have applications in cataloguing information on dye recipes.

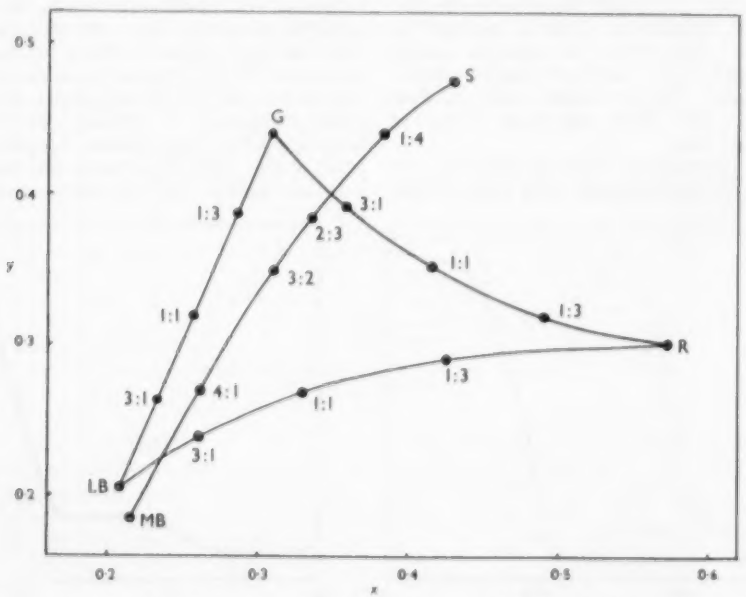


FIG. 16—Chromaticity Loci of Two-component Blends

SELECTION OF COLOURED FIBRES FOR BLENDING

If a colour catalogue is not available, it is necessary to select suitable components for the blend by other means. Fig. 16 shows curves connecting plots of components, the intermediate points being labelled according to blend composition. The blends lie on curves connecting the colours in the C.I.E. diagram, and these curves are not very far from straight lines. It is thus fairly easy to choose suitable components for a given blend, and a three-component mixing would be plotted inside a triangle whose sides linked up the components. It is not possible to ignore lightness completely, and ideally a colour solid is necessary, when four components would be at the corners of a tetrahedron.

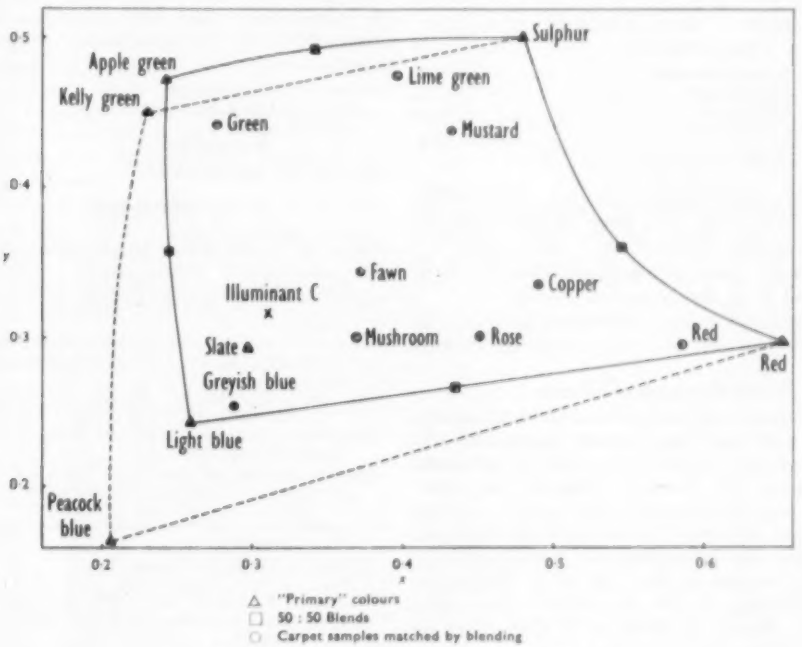


FIG. 17—Matching of Colours by Fibre Blending

With the quantitative plotting of colours in a colour solid, a small number of colours can be selected which, when mixed in various proportions, will enable a large number of colours to be matched. The "basic" colours plotted in Fig. 17 enclose an area which contains plots of colours which can be matched by blending. It follows that, if a number of colours are to be chosen as a basic range from which other colours will be matched by blending, then these colours should enclose as large an area as possible: i.e. the colours should be as saturated as possible, since blending produces a mixture which will tend to be nearer to grey than the components in a blend.

If colours are plotted on a C.I.E. diagram which has been corrected so that unit distance at any part of the diagram represents the same subjective visual change, then the basic colours may be selected so that they are uniformly spaced round the circumference of the enclosed area. Since lightness cannot be neglected, it is desirable to select the basic colours by inspection of a colour solid. In practice, in order to obtain unbroken effects with blends of coloured fibres, it is desirable to include a grey and possibly a brown in the basic range in addition to the saturated colours. This avoids the necessity of blending coloured fibres which may be well away from the grey point and on opposite sides of it.

BLEND TOLERANCES

In the production of blend yarns variations in the composition of the blend may occur. It is desirable to know the tolerances which may be permitted, e.g. whether a 50:50 blend of two colours can be allowed to drift from 49:51 to 51:49 (a variation of $\pm 1\%$) without being detected visually. The number of possible combinations in blends of Fibro-Duracol starting from eighteen basic colours is very large, and a purely empirical approach by spinning yarns with small blend variations would be tedious.

It is possible to tackle this problem by using a quantitative colour approach. Colour differences arising from 1% variations in various blends may be calculated from the reflectance curves of the separate colours.

In Fig. 18 ΔE is the calculated colour difference for a blend variation of $\pm 1\%$, i.e. an overall variation of 2%. If the 80:20 white–black blend varies from 81:19 to 79:21, the ΔE value is 0·45, whereas the corresponding figure for light blue–slate is 0·11. If we assume that the eye can detect $\Delta E = 0{\cdot}3$, then the white–black blend would

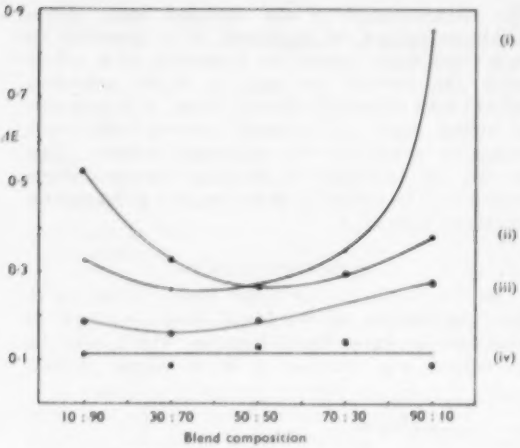


FIG. 18—Effect of Blend Variation on Colour

require close control during spinning, whereas the light blue-slate could vary from 81 : 19 to 79 : 21 without any change being visually detectable.

The curve for black-white shows that the least critical blend is in the region of 65 : 35.

These calculated colour differences have so far not been verified over the whole range of blends.

Some Subjective Effects

If the red spot in Fig. 19 is looked at continuously for 25 sec., and then the eyes are focused on the black spot on the white background, a greenish-blue afterimage will be seen. The eye has been fatigued by red, and sees the complementary colour with increased intensity. If the eye is fatigued by other colours, the corresponding complementary colours may be seen in the same way. Hence, in colour matching it is important that the surroundings should be white or neutral grey, to avoid this effect.

Effects due to the juxtaposition of colours may also be simply demonstrated. Some excellent examples have been provided by Warburton and Lund[33]. An appreciation of these phenomena is desirable in fabric design for colour and weave effects.

Concluding Remarks

It is hoped that this survey has not given the impression on the one hand that colour physics is a subject too remote from practical colour work in textiles, or on the other hand that it is approaching a position where the skill of the dyer and the practical colour worker can be dispensed with. It has been necessary to gloss over some of the difficulties and the mathematical aspects of colorimetry.

The dyer is not likely to be replaced by instrumental measurements, but he can expect some assistance, which could cut down the number of trial dyeings which are necessary at present. For long runs of a given colour, quality control is possible. We are finding applications of colour measurement rewarding in the blending of coloured fibres, and most of our own work has been in this field.

* * *

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Levelling Problems in Vat Dyeing

H. MUSSHOFF

Meeting of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 13th November 1956, Mr. W. G. B. Grant in the chair

An important factor in the poor levelling of certain vat dyes is slow migration due to molecular aggregation (up to 3000 molecules) of the leuco compound in solution. Migration may be improved by addition to the dyebath of a suitable non-ionic solvent, e.g. Solidegal GL (CFM). Practical methods for overcoming levelling difficulties in package dyeing and piece dyeing, notably the vat-acid and pad–steam processes, are discussed.

It is a well known fact that the levelling properties of the different groups of vat dyes are by no means the same. The dyer knows by experience that in general the IK group levels very well, the IW group levels well, and the IN group levels badly. Beyond this, very little is known about the factors determining the levelling properties of vat dyes and the reactions taking place during dyeing. The reason for the differing behaviour of different vat dyes may, for all practical purposes, be explained as follows—

Dyeing with vat dyes takes place in two stages: absorption and migration.

Rate of absorption is very high, even with slow-exhausting dyes, high enough, in fact, to give unlevel dyeings in package machines. Absorption can be slowed down by lowering the temperature of dyeing or by using retarding agents. But even rapidly exhausting dyes may be good levellers, provided that they migrate well enough. Migration, therefore, is the most important problem. Higher temperature as well as certain auxiliary products increase the migration properties of vat dyes.

Another decisive factor, hitherto very little known, for the levelling properties of a vat dye is the aggregation of its molecules after vatting.

When a dye is vatted, a true solution is formed only with dyes of simple chemical constitution. With indigo and thioindigo molecular solutions are obtainable. Derivatives of these dyes, however, tend to form aggregates by agglomeration of several molecules. The more complicated the chemical structure of a vat dye and the larger its molecular size, the greater is the tendency to combine and form larger aggregates. The large molecular aggregates are far less mobile than the smaller ones.

During dyeing, the dye molecule has to enter the intercellular spaces of the fibre in which it has to be fixed. If we take the swelling of the cellulose fibre during the wet treatment into consideration, the diameter of the fibre channels varies between 20 A. and 40 A. Single molecules of the IK, IW, and IN dyes are respectively 5–10, 10–20, and 20–40 A. in diameter.

In a unimolecular state, even the IN dyes would be able to enter the fibre channels, but unfortunately they agglomerate markedly and form aggregates of up to 3,000 molecules. Of course, not the whole of the dye is present in such an aggregated form in the vat: there are always a certain number of single molecules besides smaller or larger aggregates. Aggregation increases in the presence of electrolytes and at lower temperatures; it decreases in the presence of certain solvents and at higher temperatures.

While the smaller aggregates are diffusing into the fibre, the larger ones split up and form more of the small aggregates. Theoretically, a level dyeing should thus be obtained under normal conditions, but the disaggregation takes place very slowly, and it would take many hours for ideal conditions to be reached. Raising the temperature to 80–90°C. helps appreciably, but the breakdown of the large aggregates can be accelerated only comparatively slightly. Temperatures above the boiling point would speed up the process to such an extent that complete levelness would be obtained within 1–2 hr. But most vat dyes do not withstand high temperatures (110–130°C., 230–265°F.), and research has not yet been able to find chemicals which prevent overreduction, chemical re-orientation, saponification, or the splitting-off of halogen groups. There are certain methods for applying a few vat dyes at temperatures above the boil, but not nearly enough to meet all requirements.

From a consideration of the above facts it becomes understandable that certain difficulties arise in dyeing with a number of vat dyes. Ways and means have been found to eliminate these difficulties to such a degree that the practical application of vat dyes is possible without fear of faulty dyeings. There are three possible methods for overcoming dyeing difficulties—

(1) Eliminating the affinity of the dyes by incorporating them into the material in a non-substantive form, i.e. pigment or vat acid, followed by reduction and fixation *in situ*.

(2) Decreasing the rate of absorption of the sodium leuco compounds by working at low temperatures or by the use of retarding agents.

(3) Increasing the migration properties of the sodium leuco compounds by working at the highest possible temperature the dyes can withstand, and by using an auxiliary product which splits up the aggregates and thus speeds up the migration.

Up to the present time solvents have been found to be the only class of chemicals capable of appreciably increasing the migration properties of vat dyes, 5–10% solutions of pyridine or 20–25% solutions of methylated spirits being required to produce marked effects [1]. Such quantities are too expensive, and no practical solution of the problem was found until the discovery of Solidegal GL

(CFM), a non-ionic solvent. Solutions of 0·2–1·0% Solidegal GL increase the migration properties considerably without retarding the vat dyes.

PACKAGE DYEING

Besides the conventional method of package dyeing with vat dyes which level well, three useful dyeing methods can be recommended, notably in cases where difficulties cannot be overcome by the use of levelling agents alone—

Pigmentation followed by Reduction and Fixation in situ

Here it is important that the pigmentation bath exhausts well and only a very small amount of pigment remains in the bath before the second stage of the process, viz. reduction and fixation, begins. The dye pigment has to be of a particle size which is small enough to penetrate the package evenly without being filtered out by the inner or outer layers of the package, but large enough to remain inside the package. Generally, level dyeings are obtained by this method, which may especially be recommended for spun rayon staple packages, as pigmentation and reduction can take place at a higher temperature (70–90°c.), since spun-rayon packages are easier to penetrate in hot baths than in cool ones.

Levelling can be further improved by using Solidegal GL, which speeds up migration by splitting up the molecular aggregates of the vat dyes. Since the product does not disturb the vatting of the dye, it can be added to the pigmentation bath, where it improves the dispersion. Retarding agents, on the other hand, must not be employed before the dye has been completely reduced.

Temperature Control Process

Since the rate of absorption can be lowered by cooling, dyeing is started at 16–18°c. in a bath containing the vatted dye plus the necessary amount of caustic soda and hydrosulphite. This low temperature has to be kept constant during 15–20 min. Several vat dyes exhaust fairly fast even at 18°c. The temperature is then gradually raised to 35°c. at a rate of 1°c./min., and from then on at a rate of 2°c./min. until the normal dyeing temperature is reached. With those dyes which can withstand it, a temperature of 80° or even 90°c. is advisable. Solidegal GL improves levelling further. After reaching the final temperature, dyeing is continued for 20–45 min. according to the migration properties of the vat dye employed.

This process is not suitable for rayon unless the winding of the cheeses is very loose, since the swelling of the fibre in cold alkaline media prevents penetration right from the start.

Vat Acid–Temperature Control Process

In the most difficult cases, where pale or medium depths have to be dyed with badly levelling vat dyes and absolutely level dyeings must be obtained, the combined vat acid–temperature control process with addition of Solidegal GL is the answer.

The combined process replaces the earlier vat acid process, in which the vat acid was gradually transformed into the substantive sodium leuco compound by adding caustic soda and hydrosulphite in portions of $\frac{1}{5}$, $\frac{2}{5}$, $\frac{1}{5}$, and $\frac{1}{5}$ of the total amount necessary for a normal vat dyeing. The new method is safer, is easier to control, and takes less time.

The dye is vatted as stated in the pattern cards under "Stock Vats of Highest Concentration". The stock vat is added to the dyebath containing—

Acetic acid	... Sufficient to neutralise the total quantity of caustic soda used for vatting the dye
Setamol WS (BASF)	2 g./litre, as dispersing agent
Hydrosulphite	... 0·5 g./litre, to prevent oxidation of the vat acid by oxygen contained in the water
Solidegal GL	... 2–4 g./litre, as levelling agent

Dyeing is started at 16–18°c. for 15–20 min. The vat acid has no affinity for the fibre, and the package is therefore evenly penetrated with the amount of dye which corresponds to the dye liquor taken up by the package, i.e. in a liquor ratio of 10:1 one-tenth of the dye. The leuco acid is now transformed into the sodium leuco compound by adding the total amount of caustic soda and hydrosulphite needed for a normal vat dyeing process. While dyeing is still continued at the low temperature, the sodium leuco compound is slowly absorbed by the fibre, and when pale colours are being dyed, the dyebath exhausts within the next 15–20 min. The temperature has now to be raised to the highest value the dye can withstand, and dyeing is continued for 20–45 min.

This time-consuming process may safely be employed on cotton, where the other methods do not lead to complete levelness.

It goes without saying that the best dyeing method is of no use if the goods are not well pretreated, the packages are not properly wound, the pump does not give enough pressure so that there is no flow, etc.

PIECE DYEING

The old jig dyeing process does not satisfy modern demands, as the penetration, especially with dyes having a high rate of absorption as well as a low rate of migration, is too poor. Besides, there is another drawback, in that it is difficult to obtain uniformity of colour from batch to batch.

As far as jig dyeing is concerned, padding with finely dispersed pigments or vat acids, followed by reduction and fixation of the vat dye *in situ*, leads to perfect penetration and a solid appearance of the dyeing. Furthermore, it is easier to obtain uniformity of colour amongst different batches.

Vat-acid padding has the advantage of the dye being in the smallest particle size which is possible. This alone leads to better penetration, which is aided by a higher temperature during padding. Vat acids are in no way as sensitive to high temperatures and electrolytes as pigment dispersions can be. Another advantage of the vat acid is its property of rapid reduction and fixation. A normal-sized batch, padded with vat acid, has to run two ends on the jig compared with four to eight ends (according to the depth of colour) for

TABLE I

Indanthren Dye	*Colour Index* No.	Highest Concn. of Vat Acid (g./litre) Without Solidegal GL	With
Yellow 6GK conc. pdr. f.f. dyeing	C.I. Vat Yellow 27	66	100
Yellow GGF pdr. f.f. dyeing	C.I. Vat Yellow 10	32	60
Brilliant Orange RK pdr. f.f. dyeing	C.I. Vat Orange 3	35	75
Red FRC pdr. f.f. dyeing	—	55	90
Red Brown RR pdr. f.f. dyeing	C.I. Vat Brown 45	60	90
Bronze GC pdr. f.f. dyeing	C.I. Vat Brown 19	60	90
Grey NC pdr. f.f. dyeing	C.I. Vat Black 19	50	85
Blue RSN powder	C.I. Vat Blue 4	10	30
Blue BCS powder	C.I. Vat Blue 6	15	30

pigment-padded fabrics. Besides saving time, a dark appearance of the selvedges is prevented.

The application of vat acids in padding was limited to pale and medium depths by the difficulty of obtaining the concentrations necessary for padding. A solvent like Solidegal GL, however, increases the solubility of vat dyes to such an extent that practically all the problems of producing uncoagulated vat-acid suspensions have been eliminated, as, in general, two to three times the amount of dye per litre can be dissolved as when working without Solidegal GL (cf. Table I).

The increase in solubility depends, of course, on the original solubility of the dye; it is higher with a dye of poor solubility, and lower with a dye of good solubility.

Modern production methods and automation require more yards per man–hour. Continuous dyeing methods are the typical ones of our time and, more so, of the future. With reference to the application of vat dyes, two continuous processes are of great interest— the Standfast molten-metal process and the pad–steam process.

The Standfast molten-metal process is very well known in this country, and there is no necessity to discuss it in detail. Its greatest advantage is the possibility of changing from one colour to another without prolonged cleaning operations or stoppages of the machine. The small padding box coupled with the ease of cleaning allows short yardages to be run economically, and the production speed can be compared with that of any continuous dyeing range.

Pad–steam dyeing gains more and more ground. Different methods have been tried to shorten the process and to improve it technically as well as economically. One of the "economical improvements" was the wet-on-wet process, working fairly well on light-weight goods in pale colours but giving headaches with heavy dyeings and heavy fabrics. The only really reliable method is— pigment or vat-acid padding, intermediate drying, padding with caustic soda and hydrosulphite, steaming, and finishing.

There is no levelling problem to overcome unless the padding mangle is not properly adjusted, but this is an engineer's and not a dyer's problem. The fabric will leave the continuous range with the same appearance and degree of levelness as it has left the padder, and improvements are out of the question. The dyer, of course, has other problems to deal with— tailing, migration during intermediate drying, fixation, oxidation, etc.

Tailing may result from different causes. The pigment in the padder has no affinity, but, under the influence of the construction or pretreatment of the fabric, the hardness of the padding rollers, or the speed of the padder, water might enter the fabric faster than the pigment is able to do so, and thus the pigment accumulates in the padding box and tailing occurs. Washing-off of dye–pigment in the chemical padder may lead to tailing. Variable conditions in feeding the chemical padder, varying steaming as well as oxidising and soaping conditions produce tailing or colour variations.

Migration may be due to the dye or to drying conditions. Additions of thickeners and formic acid to the padding liquor eliminate migration to a great extent, but a regular and level flow of air in the hot flue is of the utmost importance.

Two-sidedness of the dyeing appears when there is too big a difference in the hardness of the padding-bowls, or if one side of the padded fabric is exposed to a higher temperature during drying than the other.

Quite frequently speckiness of padded goods has been observed, which has been mostly due to careless preparation of the padding liquor, use of unsuitable dyes, or aggregation of the dye in the presence of electrolytes and auxiliary products. Most of the vat dyes manufactured for padding nowadays do not need any addition of dispersing or wetting agents. Wetting agents should be used in the pretreatment of the goods and dried in; in this way good rewetting is ensured and foaming of the padding liquor is prevented. Foam on the padding liquor contains dye particles, which occasionally stick to the fabric and show specks. In several mills in the U.S.A. speckiness has been overcome by the use of Solidegal GL in the padding liquor.

Steaming is of the utmost importance. In the ager the dye has to be fully reduced and fixed within a very few seconds. If a vat dye needs too much time for reduction, insufficient time is left for fixation, and the unfixed dye washes off during the subsequent operations. Dyes, therefore, had to be carefully selected for pad–steam dyeing, and products with poor fixation properties had to be left aside. Here, too, Solidegal GL is a very helpful auxiliary product, as it increases the speed of reduction to such an extent that practically all vat dyes are reduced after 5–8 sec. steaming, provided that the temperature in the ager is not lower than 102°C. This means that one-quarter to one-third of the time during which the goods pass

through the ager is used for reduction, leaving sufficient time for fixation to be completed, while formerly one-half and more of the run through the ager was needed until reduction had taken place. The mechanism of this reaction can be explained by assuming that Solidegal GL disperses the dye and allows the alkali and the reducing agent to reach the dye particles without having to penetrate larger aggregates first. Furthermore, Solidegal GL prevents aggregation of the dye molecules after reduction, and the single molecules diffuse faster, so that fixation needs less time.

Oxidation may begin by an air passage immediately after steaming, except with dyes sensitive to alkaline oxidation. In most cases it is impossible to do without artificial oxidation, i.e. with air oxidation alone, since the number of washing boxes is limited and the rate of operation is mostly high. Two oxidation methods are known—oxidation with per compounds and oxidation with dichromate–acetic acid. The latter method is easier to control, but chromium residues left on the fabric may cause trouble during subsequent cutting and sewing. Peroxide oxidation gives a softer handle to the fabric, while the oxygen content of the oxidising bath needs strict control, owing to decomposition of chemicals and the evolution of oxygen.

Uniformity of colour is further influenced by the soaping process. Several vat dyes need prolonged soaping at the boil before the final colour is fully developed. If, therefore, soaping conditions are not kept constant, the colour varies, and anything between the colour before soaping and that after full development may be the result. At least two boxes of the open-width washer should be employed for soaping. To overcome soaping difficulties, it is worthwhile installing a small steam-box on top of a soaping box and letting the fabric pass through the soaping box first and then through the steam-box, which, of course, does not need to be free from air. In the hot atmosphere of the steam-box the true colour develops far more rapidly than in a soaper below the boil. Practical runs have proved that vat dyes which gave difficulties in soaping could be used with such an arrangement, which is not costly to install.

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COMMUNICATIONS

Studies in Hydrogen-bond Formation
VI—The Rôle of Hydrogen Bonds in Dyeing Processes*

D. S. E. CAMPBELL, D. CATHCART, and C. H. GILES

The results of a réfractometric study of the formation of intermolecular hydrogen-bond complexes in water between aromatic compounds, including sulphonated dyes, and a variety of other reagents, including casein, used as models of fibres, are interpreted as an indication of the nature of possible hydrogen-bond complexes between dyes and fibres. Some conclusions are—(*a*) Non-ionic polar groups in dyes and other aromatic compounds, whether sulphonated or not, can form intermolecular hydrogen-bond complexes in water with a variety of organic compounds. (*b*) Hydrogen-bond formation between dyes and fibres in presence of water appears to be possible with protein fibres, nylon, cellulose acetates, Terylene, and acrylonitrile fibres, but is less likely to occur with cellulosic fibres. Suggestions are made regarding the nature and the position of the dye–fibre bonds.

Introduction

HYDROGEN BONDS IN DYEING

The nature of the adsorption forces which cause the fixation of dyes by fibres has been the subject of much study and discussion for many years, for besides its obvious technical importance it has considerable scientific interest. These forces may be classified under three headings—(*a*) physical or van der Waals, (*b*) polar, principally hydrogen bonds, and (*c*) ion-exchange. Of these (*c*) are the most readily identifiable; they occur mainly with nylon and protein fibres. The physical forces (*a*) probably operate in dyeing several types of fibres. It is the particular purpose of the present paper to indicate the possible nature of the polar hydrogen-bond forces between dyes and various fibres.

The rôle of hydrogen-bond forces in dyeing has not previously been very clearly defined. (Early knowledge, particularly regarding cellulose and cellulose acetate fibres, is well summarised in Vickerstaff's book[1].) Any hydrogen bonds between dye and fibre must, of course, be formed in an aqueous medium, and one of the main difficulties in investigating the possibility of formation of such bonds has been the lack of knowledge of the reactivity of potentially hydrogen-bonding groups in dyes and fibres in aqueous solutions, most of the detection methods hitherto used† being applicable mainly to non-aqueous systems.

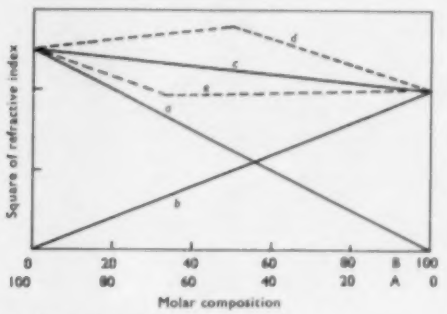
USE OF REFRACTOMETRY

Measurement of the refractive index of solutions has now been shown to be a simple means of detecting hydrogen-bond complexes between two

* Parts I–V [6–10].

† For example, radiation methods (electron and X-ray diffraction, and infrared spectrophotometry); and measurements of other properties, e.g. b.p., solubility, etc. Hunter [2] gives a useful review of these methods. For fuller descriptions of the occurrence and properties of hydrogen bonds, see references [3–5].

solutes in aqueous or non-aqueous solution, even if they have quite weak affinity for each other, and it has been applied to a large number of solutions of binary mixtures [6–10]. The results have also been checked in a number of cases by experiments with monolayers on water [11, 12].



(a) Solutions of A alone
(b) Solutions of B alone
(c) Binary solutions of A and B if no complex is formed
(d) Binary solutions of A and B if a complex AB* is formed
(e) Binary solutions of A and B if a complex A_2B* complex is formed

* The inflections in curves (d) and (e) may be either maxima or minima according to circumstances.

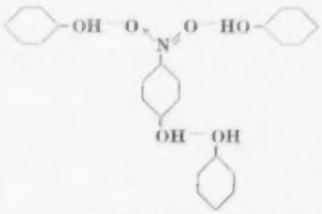
FIG. 1—Theoretical Curves showing Relation between Refractive Index and Component Ratio in Solutions of A and B

The method consists in plotting the refractive index† of solutions of binary mixtures of constant total molarity in which the relative proportions of the two components are varied over the range 0:100 to 100:0. If no complex is formed a straight line is obtained‡, because the square of the refractive index† of dilute solutions is proportional to their concentration, and is additive for mixtures of compounds if no interaction occurs (see Fig. 1). If the two components form a molecular complex, then the plot is a curve which may have a maximum or a minimum at a point corresponding with the ratio of the two components in the complex (cf. Fig. 1). In practice, the curve approximates to two straight lines intersecting at the complex-ratio point. The mathematical theory is discussed elsewhere by Eaton [13]. In systems in which more than one complex exists, each may be represented by its corresponding maximum or minimum (cf. Fig. 4 and 5).

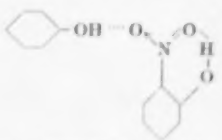
The earliest use of this principle in detecting complexes was apparently made by Ostromisslensky (1911) [14], who used spectrophotometry, not refractometry, to detect the molar ratio of organic complexes in solution. The general principles were more fully discussed independently by Denison (1912) [15]; and the spectrophotometric method has been used by other investigators (e.g. Job and collaborators [16], Vosburgh and Cooper [17]); other authors have since used measurements of refractive index (Spacu and Popper [18]; Pushin and collaborators [19]) or dielectric constant (Laurent *et al.*[20]) in a similar manner.

It has already been shown [7] that, where the results of complex detection by the present method can be checked against those of older-established procedures, the agreement is good. Where a complex is detected, it is usually possible to specify with reasonable confidence which groups are interacting, by inspection of the formulae of the components. The presence of a complex in aqueous solution means, of course, that the groups interacting can do so even in presence of water, which will itself compete by combining with each group by hydrogen bonding.

It is found that even complexes which have quite low stability are readily detected. Some indications of the presence of intramolecular chelation may also be obtained in many cases; e.g. *p*-nitrophenol is found to form a complex (I) with three molecules of phenol, but *o*-nitrophenol with only one molecule of phenol (II).



I—*p*-Nitrophenol–Phenol Complex



II—*o*-Nitrophenol–Phenol Complex

PRESENT WORK

The method is used in the present investigation to study the interaction in water of typical dyes or compounds containing groups present in dyes, with "models" of fibrous polymers. Thus glucose and sucrose are used as models for cellulose, ethyl acetate and glycerol triacetate for cellulose acetate, simple amides and also casein for protein fibres, and acetonitrile for polyacrylonitrile.

In the subsequent discussion it is assumed that, if groups in any of the pairs of solutes used do not interact by hydrogen bonding in aqueous solution, they would not do so if the one were present in a fibre and the other in a dye. The conclusion from an opposite result is not so definite. If two groups do interact in the present tests, it does not always follow that their presence in dye and fibre respectively will cause the dye to be substantive, for their interaction may not be strong enough to break down internal bonds in the fibre itself. If, however, the dye is in fact found to be substantive to the appropriate fibre, then it appears reasonable to assume that at least some interaction of the type detected in solution is occurring also between fibre and dye. These points are discussed more fully below.

† Strictly the *square* of the refractive index should be used to give a linear relation with concentration, but in practice the differences are usually so small that the index itself, or even (in the case of the Pulfrich instrument) measurement of the angle of the emergent beam, gives equally good results, and their use avoids tedious computations.

‡ Conversely, however, a linear plot does not necessarily indicate conclusively that a complex cannot be formed *cf.* [7].

TABLE I

Detection of Complexes by Refractive Index Measurement

Solutes (*a*)	(*b*)	Total Molar Concn.	Mol. Ratio of Complex (*a* : *b*)	Fig. No.
	DYES AND OTHER AROMATIC COMPOUNDS IN WATER			
Ethanol	C.I. Acid Orange 8	0·05	—[a,b]	—
Ethylene glycol	C.I. Acid Orange 8	0·05	—[a,b]	—
	C.I. Acid Orange 7	0·1	—[a,b]	—
Dimethylformamide	F acid	0·1	1 : 1†	—
	ε acid	0·1	1 : 1†	—
	C.I. Acid Orange 20	0·1	2 : 1‡	—
	C.I. Acid Orange 7	0·1	(1 : 1 or 1 : 2)(?)	—
	C.I. Acid Red 1	0·1	2 : 1	—
	C.I. Acid Blue 47	0·01	1 : 1‡ (?)	—
	C.I. Acid Blue 45	0·1	1 : 1, 2 : 1‡	—
N-Methylacetamide	C.I. Acid Orange 7	0·1	1 : 1(?), 2 : 1	—
	C.I. Acid Red 1	0·1	2 : 1	—
	Anthraquinone-2-sulphonic acid	0·1	2 : 1	—
Phenol	F acid	0·1	1 : 1†	—
	ε acid	0·1	1 : 1†	—
	Anthraquinone-2-sulphonic acid	0·1	2 : 1	—
	Naphthalene-1-sulphonic acid	0·1	—[a]	—
	R acid	0·1	1 : 1	—
	Amino-R acid	0·1	—[a]	—
	C.I. Acid Orange 20	0·1	3 : 1, 1 : 1‡	—
	C.I. Acid Orange 20	0·25	1 : 1, 2 : 1, 3 : 1	—
	C.I. Acid Orange 7	0·1, 0·25	1 : 1	—
	C.I. Acid Orange 8	0·05	1 : 1	—
	C.I. Acid Red 1	0·1	2 : 1	—
	C.I. Acid Blue 47	0·01	1 : 1‡	5
	C.I. Acid Blue 45	0·1	1 : 1, 2 : 1‡	5
Pyridine	C.I. Acid Red 1	0·1	1 : 1	—
Quinol	C.I. Acid Orange 7	0·1	1 : 2[c]	—
	CARBOHYDRATES IN WATER			
D-Cellobiose	Dimethylformamide	0·25	1 : 1[b]	—
	Pyridine	0·1	1 : 1[b]	—
D-Fructose	Phenol	0·25	—[a]	—
D-Glucose	Aniline	0·25	2 : 1	—
	Dimethylformamide	0·25	1 : 1[b]	—
	Phenol	0·25	—[a,b]	—
	Pyridine	0·25	1 : 1[b] (?)	—
	Quinol	0·25	—[a,b]	—
	C.I. Acid Orange 7	0·1	1 : 1	3
	C.I. Acid Red 1	0·1	2 : 1	—
Sucrose	C.I. Acid Orange 7	0·1	—[a]	3
	C.I. Acid Red 1	0·1	—[a]	—
	Dimethylformamide	0·1	—[a]	—
	ACETATES AND ALDEHYDES IN WATER			
Ethyl acetate	4-Hydroxyazobenzene-4′-sulphonic acid	0·1	3 : 1 (?)	—
	p-Nitrophenol sulphuric ester	0·04	2 : 1	—
	C.I. Acid Orange 7	0·1	1 : 2 (?)	—
Glycerol triacetate	4-Hydroxyazobenzene-4′-sulphonic acid	0·025	—[a]	—
	p-Nitrophenol sulphuric ester	0·04	1 : 1	—
Propionaldehyde	Aniline	0·1	2 : 1	—
	C.I. Acid Orange 7	0·1	1 : 1	—
	ETHER GROUPS IN WATER			
Dioxan	Ethanol	0·1	1 : 2	—
	Phenol	0·1	(1 : 1 or 1 : 2)	—

TABLE I—*continued*

Solutes (a)	(b)	Total Molar Concn.	Mol. Ratio of Complex (a : b)	Fig. No.
	CASEIN IN pH 9 BUFFER			
Casein	C.I. Acid Orange 7	0·1	ca. 1 : 4, ca. 3 : 2	4
	C.I. Acid Red 1	0·1	ca. 2 : 3, 3 : 2	4
	Naphthionic acid	0·1	ca. 2 : 1	4
	Naphthalene-2-sulphonic acid	0·1	—*	—
	F acid	0·1	ca. 1 : 1	—
	Anthraquinone-2:7-disulphonic acid	0·1	—*	—
	C.I. Acid Blue 45	0·1	ca. 2 : 1, ca. 1 : 2	—
	AROMATIC ESTERS IN TOLUENE			
Phenol	Dimethyl terephthalate	0·1	4 : 1	—
	Ethylene glycol dibenzoate	0·1	4 : 1	—
Triethylamine	Dimethyl terephthalate	0·1	4 : 1	—
	Ethylene glycol dibenzoate	0·1	4 : 1	2
	NITRILE GROUP IN WATER			
Pyridine	Acetonitrile	0·1	1 : 1	2
	SALT FORMATION BY ACID DYES			
Glycine	C.I. Acid Orange 7	0·1	1 : 1†	—
Triethylamine	C.I. Acid Orange 8	0·05	1 : 2†	—

* No complex detected.
† In pH 9 buffer; in unbuffered solutions indications were less definite.
‡ Examined in suitable lines from the cadmium–mercury lamp; with all other systems sodium light was used.
For comparison, some of the data are quoted from previously published work, as indicated.

The detailed results of the present tests are set out in Table I, a few typical graphs are shown in Fig. 2–5, and the implications are discussed below.

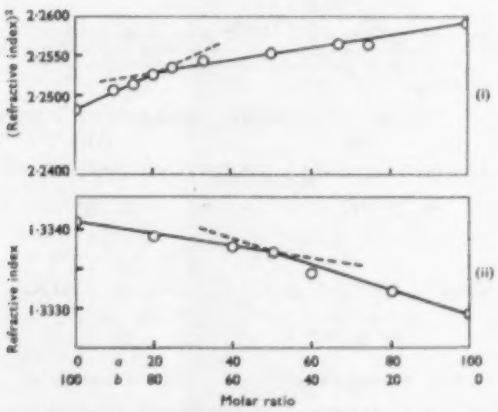
Experimental

REAGENTS

The dyes were purified to 95–98% purity by recrystallisation from water or ethanol–water mixture. The casein was of "light white soluble" quality (B.D.H.). Other reagents were either of commercial analytical reagent quality or were purified by normal methods. Distilled water was used for all aqueous solutions. Toluene was dried over sodium.

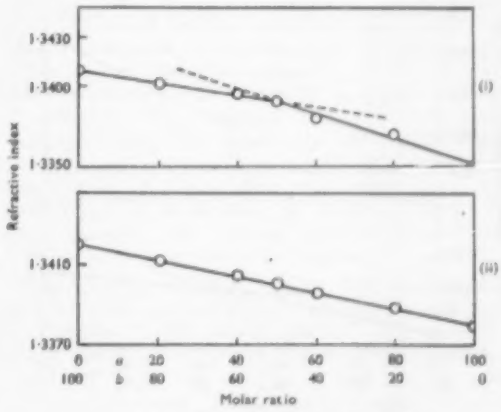
APPARATUS

The Zeiss Abbe and the Bellingham & Stanley Pulfrich refractometers were used, at room temperature, and normally with sodium light. Where the solutions were not sufficiently transparent to this light, suitable lines from the mercury–cadmium lamp were used. It is found to be advisable in some cases to allow the solutions to stand, tightly stoppered, for several hours or overnight to reach equilibrium before measurement.



(i) (*a*) Ethylene glycol dibenzoate, (*b*) triethylamine, in toluene
(ii) (*a*) Pyridine, (*b*) acetonitrile, in water

FIG. 2— Complex Formation by Esters and Ethers



(i) (*a*) D-Glucose, (*b*) C.I. Acid Orange 7
(ii) (*a*) Sucrose, (*b*) C.I. Acid Orange 7

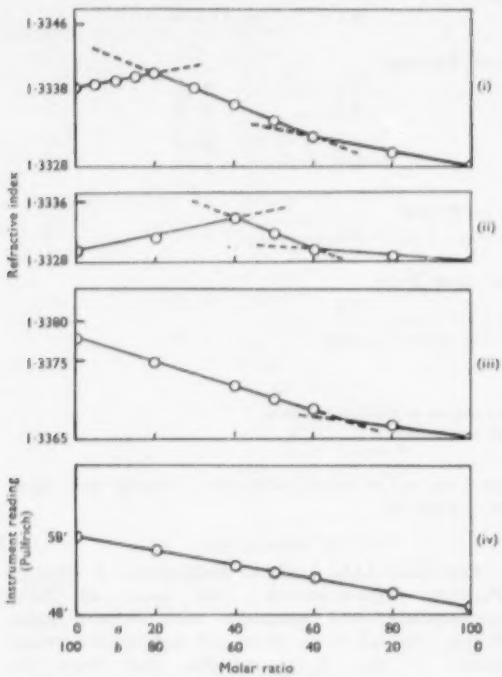
FIG. 3— Complex Formation by Glucose and Sucrose in Water

Results and Discussion

HYDROGEN-BONDING PROPERTIES OF DYES

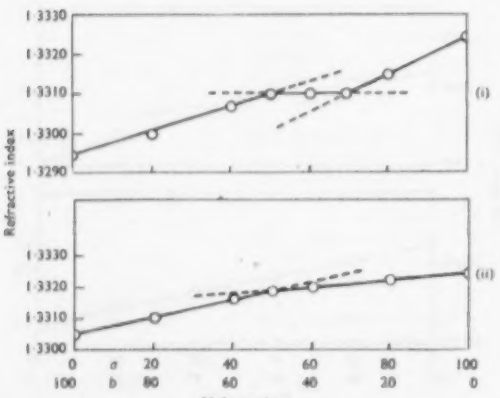
(a) Complexes formed by Sulphonated Compounds with Hydrogen attached to Nitrogen or Oxygen

One important conclusion from these and some of the previously reported results [7] is that sulphonation of a molecule, with the consequent high degree of solvation in water, does not impair its ability to form many intermolecular hydrogen-bond complexes in aqueous solution. Thus, the phenolic groups in ε acid, F acid, or Orange II appear to be reactive towards dimethylformamide, *N*-methylacetamide, or phenol. Alcohols, however, do not appear to interact with sulphonated dyes (cf. Table I).



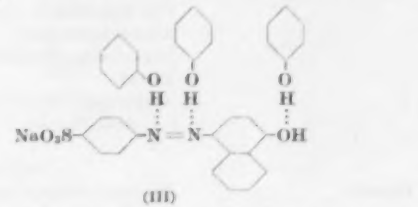
(i) (*a*) Casein, (*b*) C.I. Acid Orange 7
(ii) (*a*) Casein, (*b*) C.I. Acid Red 1
(iii) (*a*) Casein, (*b*) naphthionic acid
(iv) (*a*) Casein, (*b*) naphthalene-2-sulphonic acid

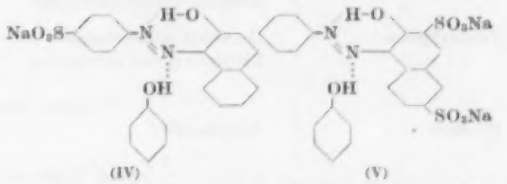
FIG. 4— Complex Formation by Casein in pH 9 Buffer



(i) (*a*) Phenol, (*b*) C.I. Acid Blue 45
(ii) (*a*) Phenol, (*b*) C.I. Acid Blue 47

FIG. 5— Complex Formation by Dyes in Water

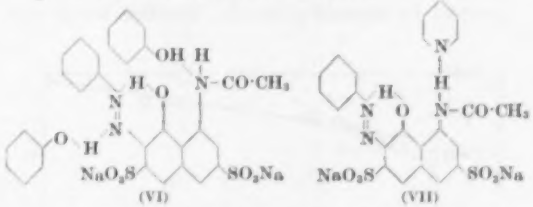




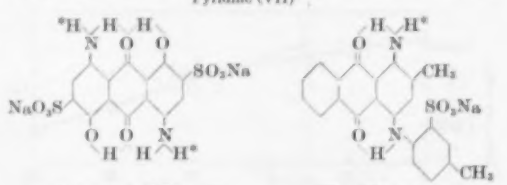
Suggested Formulae of Complexes of Phenol with Orange I (C.I. Acid Orange 20) (III), Orange II (C.I. Acid Orange 7) (IV), and Orange R (C.I. Acid Orange 8) (V)

It is clear that the three reactive centres in Orange I can act independently, and this dye can form complexes with either three or two phenol molecules or even a single one. Presumably in the last-named case the two nitrogen atoms are unreactive. (The unsulphonated compound (aniline→*α*-naphthol) gives the corresponding three complexes with phenol in benzene solution [7], so that the phenolic groups and the azo group are interacting in the same way in non-aqueous as in aqueous solution.)

The presence of a chelate ring in Orange II and Orange R is indicated by their combination with only one molecule of phenol (IV and V). In C.I. Acid Red 1 there must also be a chelate ring, and the complexes it forms with phenol and pyridine may be formulated as VI and VII.



Suggested Complexes of C.I. Acid Red 1† with Phenol (VI) and Pyridine (VII)



VIII— C.I. Acid Blue 45‡ IX— C.I. Acid Blue 47

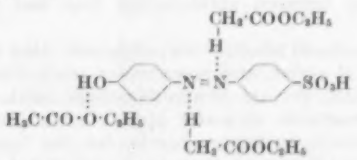
† Alternatively, the hydroxy group in the 1-position may chelate with the nitrogen atom of the acetamido group in position 8, but this would not alter the component-ratio in the complex.

‡ Cf. 1:8-dihydroxyanthraquinone, which has one carbonyl oxygen atom included in two chelate rings (Pauling [8]).

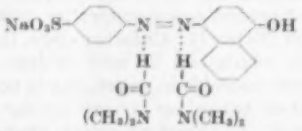
The two anthraquinone blues (VIII, IX) give complexes which are consistent with the chelated structures shown, the hydrogen atoms marked * on the amino groups remaining capable of intermolecular complex-formation. Thus C.I. Acid Blue 45 can form a 1:2-complex (also a 1:1-complex) with phenol or dimethylformamide; the other dye forms only 1:1-complexes (Fig. 5).

(*b*) *Complexes formed with Dyes through Hydrogen attached to Carbon*†

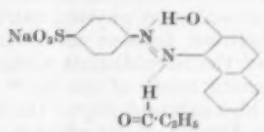
A hydrogen atom attached to carbon forms hydrogen bonds less readily than one attached to oxygen or nitrogen, but it can do so if activated by strongly electronegative groups, and such bonding, e.g. by the hydrogen in chloroform, is well known. Similarly, a carbonyl oxygen atom can activate a hydrogen attached to a carbon atom, and it has been suggested that by this means the aldehyde group and certain ester groups can act in water as proton-donors, towards nitrogen atoms in a second solute [10]. Bonds of this nature may account for several complexes detected here, e.g. X–XII. Glucose in water behaves as the open-chain aldehyde [9] (cf. its reactions with those of propionaldehyde—Table I), and appears to form –CH... bonds with some proton-acceptors. Its 1:1-complex with Orange II and its 2:1-complex with C.I. Acid Red 1 may, perhaps, be formed in this way. In the last-mentioned dye the most likely positions for attachment of a –C–H... bond are the two nitrogen atoms with which phenol is probably combined (cf. VI). The 2:1-complex between dimethylformamide and the same dye may be of a similar nature, the alkylamide being combined through a –C–H... group. Other complexes in which this bond may, perhaps, be present are shown below (X–XII).



X—Suggested Structure of Ethyl Acetate–4-Hydroxyazobenzene-4'-sulphonic Acid Complex (the phenolic group is assumed attached to the ether oxygen in the ester, for reasons discussed below)



XI—Suggested Structure of Dimethylformamide–Orange I Complex



XII—Suggested Structure of Propionaldehyde–Orange II Complex

† CH... bonds with aromatic nuclei have been reported several times, but are not considered here.

HYDROGEN-BONDING PROPERTIES OF CASEIN

Casein was used here as a model for protein fibres‡. It dissolves readily in pH 9 buffer, use of this alkaline buffer being necessary to prevent salt formation between amino groups in the protein and sulphonate groups in the aromatic solute. Such salts would probably themselves register as complexes by the present method (cf. the glycine–Orange II and triethylamine–Orange R complexes (Table I), which must be amine–sulphonic acid salts). For purposes of calculation the protein is assumed to be a monomer with the average molecular weight (125) of its constituent amino-acid residues [21]. Thus if a second solute unites with every peptide linkage, it should appear to form a complex of approximately 1:1-ratio. Results are shown in Table I and Fig. 4. There is evidence of intermolecular association between the protein and all the aromatic compounds containing potentially hydrogen-bonding groups. There is no evidence of bonding between casein and naphthalenesulphonic acid, which would not, of course, be expected to form a hydrogen bond. It is not possible, however, from the limited evidence here, to formulate the precise structure of the complexes formed.

HYDROGEN-BONDING PROPERTIES OF FIBRES

Cellulose

This fibre can probably adsorb solutes by hydrogen bonds either when the solution is highly concentrated (e.g. urea from water [22]) or when water is present only in very low concentration (e.g. acid dyes in 95% ethanol [23] or phenol in *iso*octane [24]). In normal dyeing processes, however, dye–cellulose hydrogen bonds are probably of much less importance than non-polar forces, because water is so strongly attached to the glucose residues of the fibre that it is displaced only with great difficulty by any solute [7, 10, 11]. Phenol, for example, is not adsorbed by cellulose from dilute aqueous solutions [24, 25], whereas it is strongly adsorbed by most other fibres. It follows that physical or van der Waals forces are probably the principal cause of attraction between cellulose and dyes. The need for dyes for cotton to have long and planar structures, first pointed out by Hodgson [26], arises in the great enhancement of intermolecular force produced by the extended conjugate system so formed. Thus Coulson and Davies [27] have calculated that the interaction energy between pairs of long conjugated molecules of the polyene series must vary as about the third power of their length for separations of about 4 A., which is approximately the distance that may separate cellulose and dye molecules in water.

The inability of dyes to form hydrogen bonds with cellulose appears to be confirmed by some of the present results with glucose and sucrose. These results give no evidence that the hydroxy groups of these two compounds can form bonds with dyes in water. It appears that sucrose forms no complexes at all with sulphonated dyes, and

‡ Gelatin in neutral aqueous solution also gave indications of hydrogen bonding with dyes.

glucose, as discussed above, forms them only through its aldehyde group. The glucose rings of the chain molecules in the amorphous regions of cellulose are in a state of solution in water [28], and though they have, of course, a lower average number of hydroxy groups per glucose unit than glucose or cellobiose themselves have, it seems likely that they must be protected in a similar manner against hydrogen bonding by their water "atmosphere". Possibly this protection of carbohydrate molecules in aqueous media serves some essential purpose in biological systems.

Peters and Sumner [29] have interpreted certain affinity data for vat dyes on cellulose as indicative of the operation of both van der Waals and hydrogen-bond attraction between dye and fibre in certain cases where a strong polar group, e.g. –CO·NH–, is present in the dye. This may indicate that a group of this nature can, when the non-polar forces are sufficiently powerful, displace water from cellulose, but an alternative explanation of the results may be given. This will be discussed in a later paper.

Cellulose Acetate

The mechanism by which dyes are adsorbed by this fibre, differing as it apparently does so considerably from the older-established fibres, has long been a source of interest. Vickerstaff [1] admirably summarises earlier ideas, which were largely concerned with the possibility of "solid solution" being the mechanism. Marsden and Urquhart [25] first suggested hydrogen-bond formation, following their very thorough investigation of the adsorption of phenol and related compounds by this substrate. They suggested that the phenolic hydroxy group forms a hydrogen bond with the carbonyl oxygen atom of the acetate group. This hypothesis was later extended by Vickerstaff [1] to cover the adsorption of the disperse dyes.

While most of the facts appear to support these suggestions, they do not adequately account for the adsorption of a number of dyes which are not proton-donors. Mechanisms later suggested include— (*a*) –CH... bonds formed through the methyl group in the acetate residue, activated by the adjacent –C=O group [10, 11] (this receives some support from refractometric and other physical data, including infrared spectrophotometry; the latter gave indications of changes in the $-CH_3$ and –O– groups, but not in the C=O groups, when two acetate residues were adjacent and were believed to be chelated [10]); (*b*) dipole–dipole interaction with –C–O groups [30]; and (*c*) non-polar van der Waals forces [31]. The present results support the suggestion of mechanism (*a*) in that they are consistent with complexes being formed through –CH... bonds between esters and dyes in water, as already discussed. Other relevant conclusions may also be drawn from refractometric work— thus (*a*) no definite evidence can be obtained that a C=O group in any compound can form intermolecular bonds *in water*; (*b*) in water the –CH... bond forms readily with a nitrogen atom in the second solute, but not with oxygen, e.g. in alcohols or phenols; and (*c*) a bond can form in water between phenol and an ether oxygen atom (cf. reactions with dioxan, Table I).

The following hypotheses are therefore now suggested to account for hydrogen-bond adsorption of dyes by cellulose acetates— (i) the earlier suggestions [1, 25] of the –C=O...HO–Dye bond are modified to that of a bond on the ether oxygen atom, e.g.—

$$\begin{array}{c}\text{Cellulose–O–CO–CH}_3\\ \vdots\\ \text{HO–Dye}\end{array}$$

(ii) azo or dialkylamino groups in the dye may be linked by means of a –CH... bond to their nitrogen atoms; (iii) the bond formed by unsubstituted amino groups in the dye may, perhaps, be of either of these types.

It will be seen, therefore, that most of the oxygen and nitrogen atoms in a dye can form one or other type of bond with an acetate group; moreover, since there is an abundance of such groups closely spaced in the cellulose acetate molecule, there seems no reason to doubt that all the oxygen and nitrogen atoms in a single dye molecule, except those forming part of a chelate ring, could simultaneously form hydrogen bonds with the substrate. These suggestions do not, of course, rule out the possibility that other forms of attraction may operate as well.

Protein Fibres and Nylon

The most abundant potentially hydrogen-bond-forming groups in these fibres are the –CO·NH– groups, and the present method was therefore used to detect the possibility of bonding with these. The rôle of hydrogen bonding in adsorption of acids and acid dyes by protein fibres has been discussed by Vickerstaff [1, 32]. There has previously been little direct evidence of the possibility of such bonding between sulphonated dyes and protein fibres.

The present results with casein and other models, discussed above, and more recent work (Cameron, MacEwan, private communications) with monolayer reactions on water appear to indicate that dye–protein hydrogen bonds can be formed in water*.

Terylene

Esters used as models of this polyester fibre give evidence of proton-donor properties, just as do the acetates (cf. Table I). Unfortunately, the simplest compounds which can be used as true models of this fibre are insoluble in water, but in non-aqueous solvents their behaviour is very similar to that of the acetates. It therefore seems likely that the bonding mechanism of this fibre resembles that suggested above for cellulose acetate, though the powerful intermolecular physical attraction which can exist between benzene nuclei must operate here, between the terephthalate nuclei in the fibre and the aromatic nuclei of the dye [34]. This would explain the high substantivity of the hydrocarbon, diphenyl, for Terylene [35], for which it is used as an assistant ("carrier") in dyeing.

* Elöd and Fröhlich [33] have suggested that hydrogen bonds form between acid dyes and protein fibres at the isoelectric point.

Polyacrylonitrile Fibres

In these the polar group is the –CN group, which has a powerful activating effect on an attached –CH group, leading to the formation of –CH... bonds, as e.g. in the hydrogen cyanide crystal[4]. Kumm[36] has in fact suggested that the intermolecular bond in these fibres is formed in this way. The present tests (Table I) show that a bond can be formed in water between acetonitrile and a proton acceptor, pyridine, so that the –CH... bond is likely to be formed between polyacrylonitrile fibres and nitrogen atoms in dyes. Hydroxy groups in dyes, it may be suggested, may form the –CN...HO– bond.

Conclusions

The results of this investigation may be summed up thus—

(*a*) Non-ionic polar groups in dyes and other aromatic compounds, whether sulphonated or not, can form intermolecular hydrogen-bond complexes in water with a variety of organic compounds.

(*b*) Hydrogen-bond formation between dyes and fibres in presence of water appears to be possible with protein fibres, nylon, cellulose acetates, Terylene, and polyacrylonitrile fibres, but is less likely to occur with cellulosic fibres.

Note— If a dye molecule can be adsorbed by a fibre through a hydrogen bond, it does not necessarily follow that in the dyeing process the whole of the adsorbed dye is taken up by the same process. After the first adsorption of dye in a monolayer, the remainder may preferentially become associated with the dye molecules already adsorbed, i.e. it may form aggregates, rather than be directly adsorbed on the fibre molecules.

* * *

The authors thank Professor P. D. Ritchie for his interest and encouragement, Dr. M. A. T. Rogers and Imperial Chemical Industries Ltd., Dyestuffs Division, for the gift of a number of reagents and dyes, and Imperial Chemical Industries Ltd., Central Research Department, for financial assistance in the purchase of equipment.

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The Neutralisation of Acid-treated Wool by Solutions of Sodium Carbonate

R. P. HARKER

Neutralisation of acid-treated wool by solutions of sodium carbonate has been examined by measurement of hydrogen-ion concentrations. The effects of concentration, time, liquor ratio, and temperature on the equilibration process have been studied. A qualitative interpretation of results is given, and a quantitative treatment employing the Donnan theory of membrane equilibria has been examined.

INTRODUCTION

The sorption of simple acids and bases by fibrous proteins, such as wool, has been the subject of investigation by a number of workers [1–11]. From studies of the combination of acids with wool, theoretical treatments have been advanced by Steinhardt and co-workers [4, 5], Gilbert and Rideal [6], and Peters and Speakman [7]; these theories give somewhat similar results in practice, but are based on the assumption of different physical phenomena. The theories give two distinct physical pictures, the one favouring adsorption on specific sites [6], i.e. Langmuir-type adsorption, the other preferring a model based on a Donnan-type membrane equilibrium [7]. Neither of these theories can be made to fit experimental findings entirely, and it may well be that the final solution will come from a combination of both. Recently, Horner [10] has shown that the Donnan treatment, applied to the combination of sodium hydroxide with wool keratin, gives the better agreement with experimental data.

These results are of considerable theoretical interest and, equally important, they have a direct practical significance for wet processing in the woollen and worsted industries. In a previous paper [11] the author has given a qualitative account of the absorption of alkali by wool from aqueous solutions of sodium carbonate and bicarbonate, and has attempted a quantitative treatment employing the Donnan theory.

The present work is concerned with a qualitative picture of the absorption of sulphuric acid by wool and the neutralisation of acid-treated wool by aqueous solutions of sodium carbonate.

EXPERIMENTAL

The measurement of hydrogen-ion concentration was used to follow the absorption process of both acid and alkali. The general technique applied was to equilibrate a known weight of wool (weighed at a standard condition of 16% absorption regain) in a solution of known concentration and measured pH. After equilibration for a standard time, the wool was filtered off and the pH of the equilibrium solution was measured. Where necessary, titration of the liquors before and after equilibration was carried out to determine the amount of solute absorbed from solution. The wool samples were washed quickly with a minimum quantity of distilled water and then dried in a vacuum oven at 40°C. The pH values of the aqueous extracts of these samples were subsequently determined strictly in accordance with the I.W.T.O. specification [12].

All equilibrations, except where otherwise mentioned, were carried out in a thermostat at $20 \pm 0{\cdot}1$°C. Aqueous extractions were carried out at room temperature (14–16°C.), and subsequent pH measurements were made at the same temperature.

MATERIALS

The wool samples were prepared from a bulk supply of dry-combed Australian 64s A Merino tops (scoured by a normal soap–soda process) which had been reduced to a finely divided state ($\frac{1}{64}$ in. mesh) by a Blackfriars Junior rotary cutter; this gave a material which could be handled easily and separated from excess solution by filtration under vacuum. The wool was exhaustively extracted with polar and non-polar solvents, and then washed with distilled water (adjusted to pH 5·0 with sulphuric acid) until the wash water had a steady pH of about 5.

All the chemicals used were of A.R. grade, and the distilled water had a pH of 6·0–6·4.

pH MEASUREMENTS

Measurements of pH were made on a Cambridge laboratory meter; the electrode assembly consisted of a dip-type calomel electrode and a "universal" glass electrode (Alkacid electrode: Doran Instrument Co. Ltd., Stroud, Gloucestershire). The system was calibrated in 0·05 M. potassium hydrogen phthalate solution (pH = 4·00 at t = 15°C.) and 0·05 M. borax solution (pH_t = $9{\cdot}27 - 0{\cdot}0085\,(t - 15)$). The glass electrode was calibrated in carbonate-free solutions of sodium hydroxide of known concentration. The observed pH was plotted against the value calculated from the relation—

$$\mathrm{pH} = \mathrm{p}K_w + \log [\mathrm{NaOH}] f_{\mathrm{NaOH}} \qquad \text{(i)}$$

the values for the mean activity coefficient f_{NaOH} being obtained from the data of Harned and Owen [13]: this gave a straight-line relationship for the pH range 8–13. In acid solutions of pH > 1, measured values agreed with those calculated from the mean activity coefficient for sulphuric acid [13] and also with values calculated from dissociation constants [14]. In stronger solutions, some deviations occurred: these were of the same order as those noted by other workers [5] and were probably due to activity effects in solutions of high ionic strength (e.g. pH = 0·5; ionic strength ca. 1·0) when the meter was standardised at low ionic strength (pH = 4·0).

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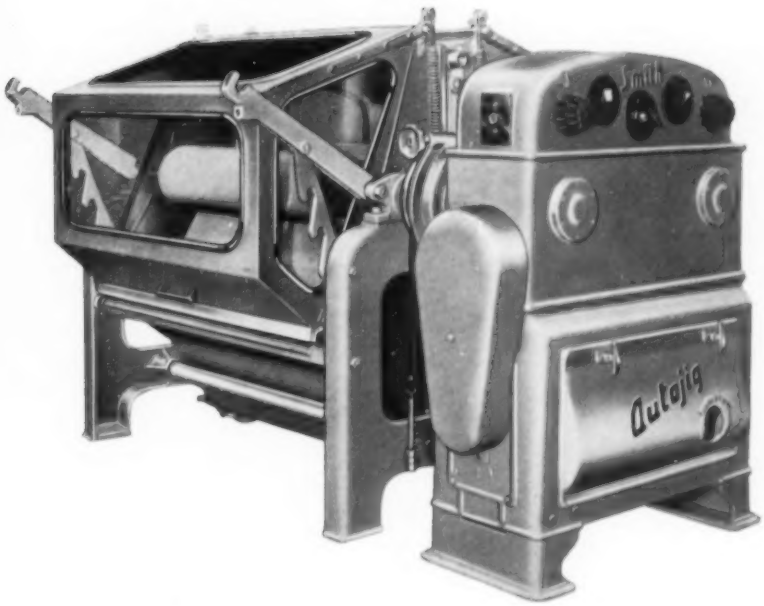

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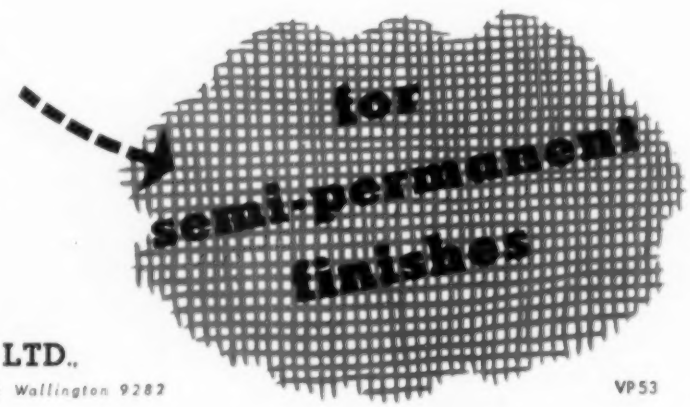

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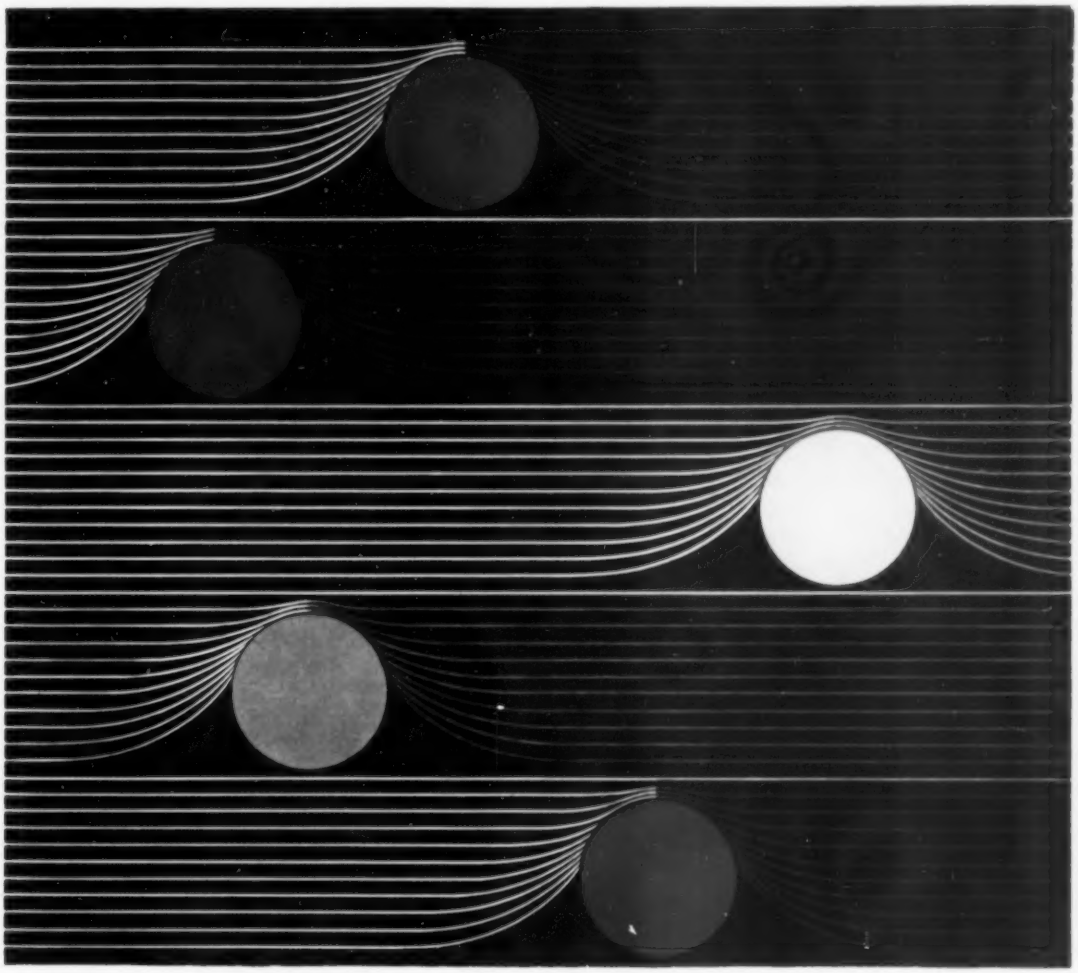

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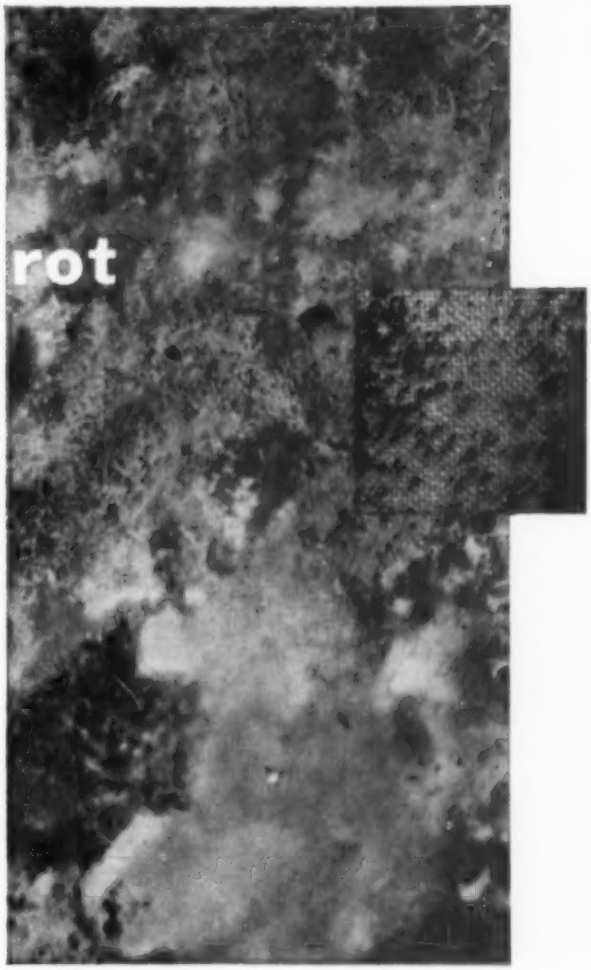

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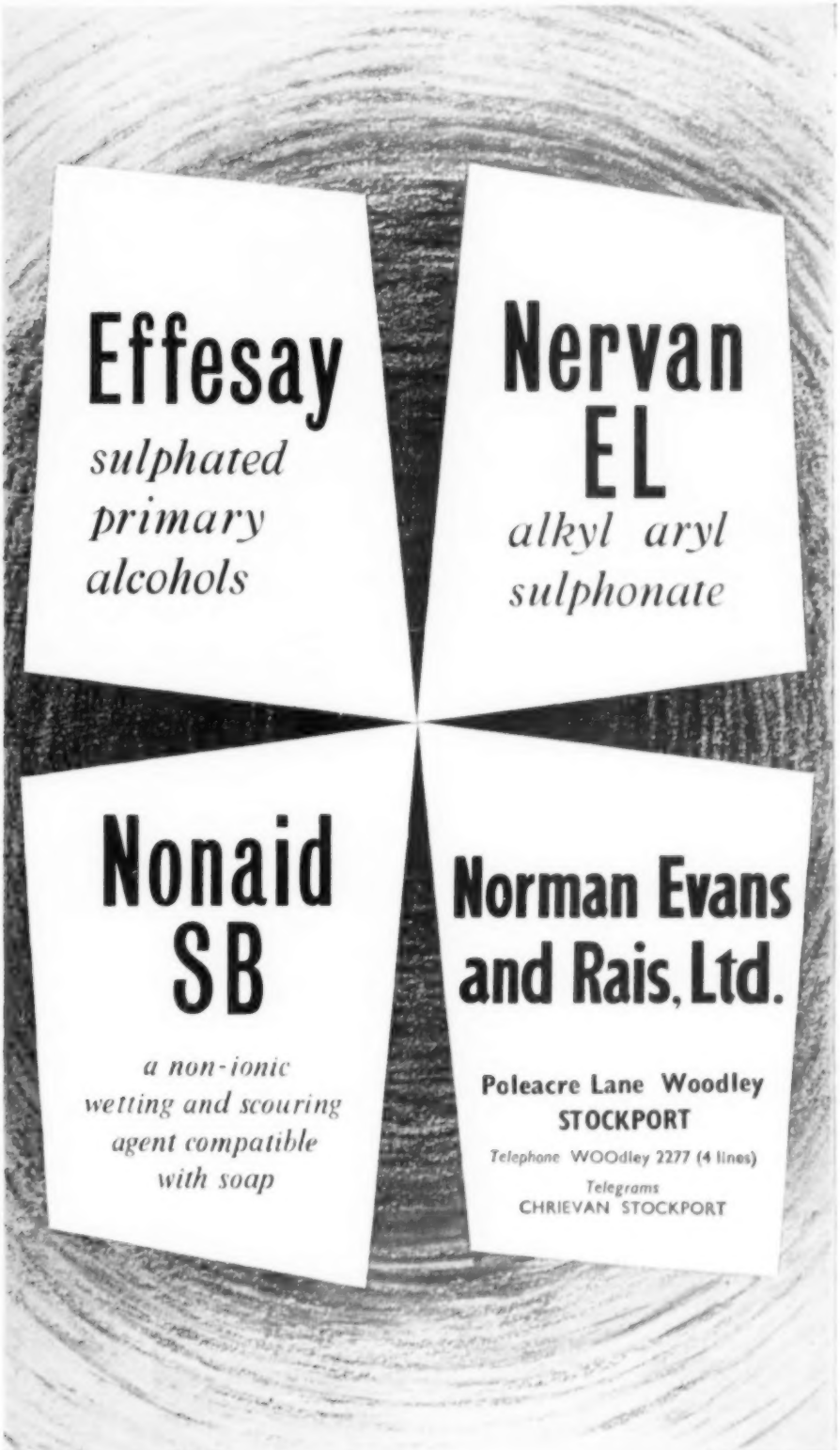
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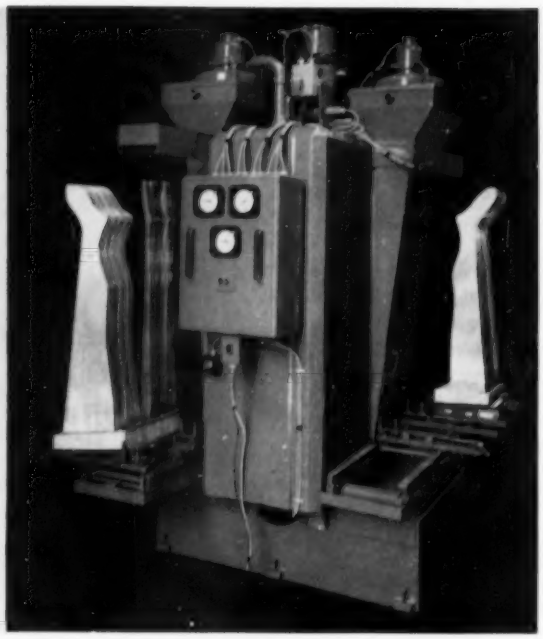

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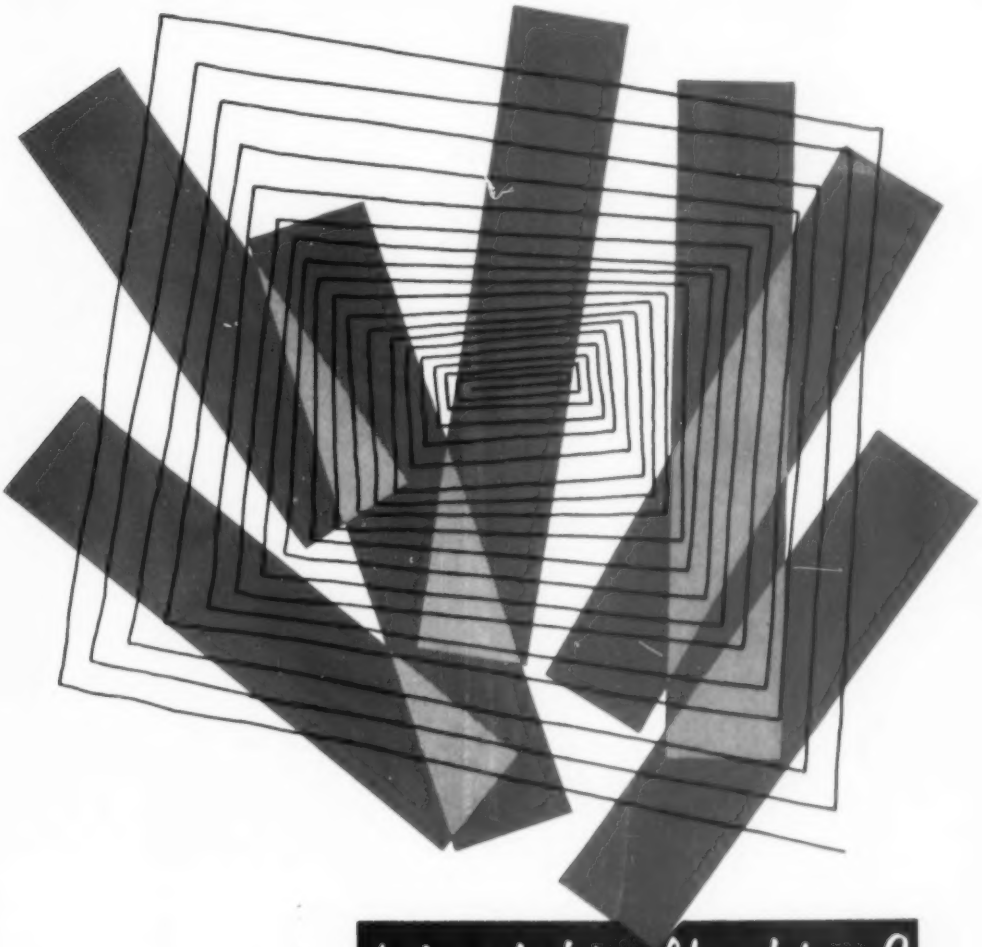

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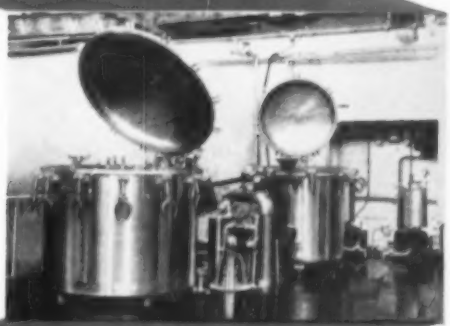

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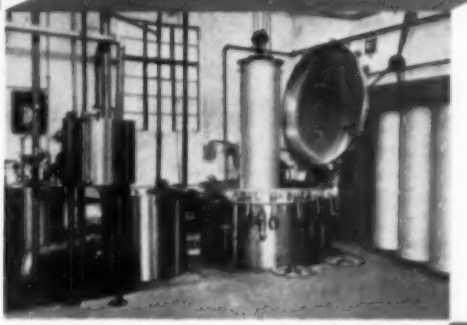

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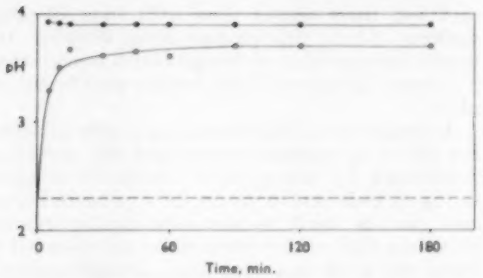

FIG. 1—Effect of Time of Equilibration (20°C., liquor ratio 20:1)

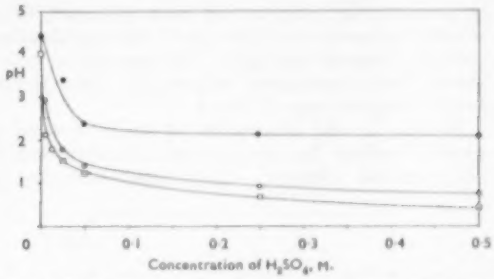

FIG. 2—Effect of Acid Concentration (20°C., liquor ratio 20:1, 60 min.)

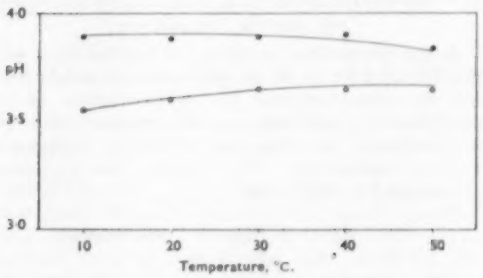

FIG. 3—Effect of Temperature (0·005 M-H_2SO_4, liquor ratio 20:1, 60 min.)

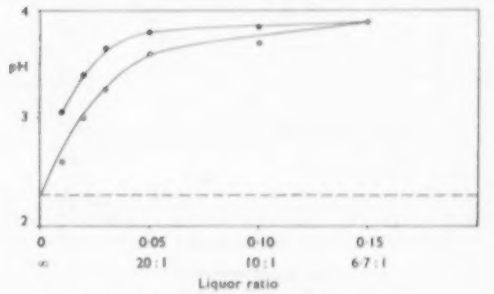

FIG. 4—Effect of Liquor Ratio (20°C., 30 min.)

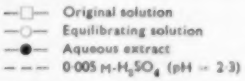

FIG. 1–4—Absorption of Sulphuric Acid by Wool

RESULTS

I. ABSORPTION OF SULPHURIC ACID BY WOOL

(i) *Effect of Time of Equilibration*

(Fig. 1)

The absorption of acid is fairly rapid, even in the case of 0·005 M. solutions, equilibrium being nearly complete after 20 min. The desorption cycle showed that for equilibration times greater than 10 min. the pH of an aqueous extract does not vary greatly.

(ii) *Effect of Concentration of Equilibrating Solution*

(Fig. 2)

In concentrations of acid greater than ca. 0·1 M. the equilibration curve follows the pH curve of the original solution very closely and the aqueous-extract curve shows that the amount of acid taken up does not vary greatly. In weaker solutions, the absorption is considerably reduced.

(iii) *Effect of Temperature*

(Fig. 3)

From these results, it would appear that slightly more acid is taken up at 50°C. than at 10°C. This is shown by the slight rise in pH of the equilibration curve with temperature and by a corresponding decrease in the slope of the desorption curve.

(iv) *Effect of Variation in Liquor Ratio*

(Fig. 4)

Where the reciprocal of the liquor ratio is zero, no wool is present, theoretically, and the equilibration curve cuts the pH axis at pH = 2·31, the original pH of the equilibrating solution. As the liquor ratio decreases and progressively less liquor

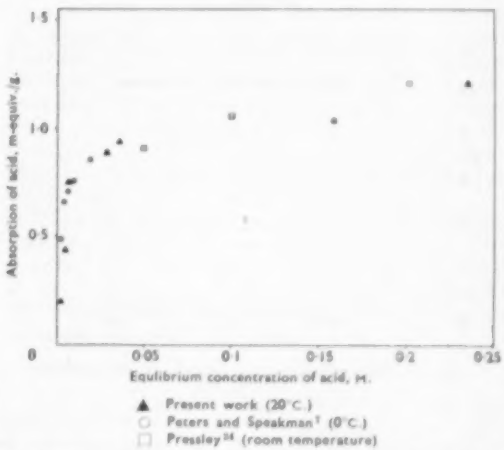

FIG. 5—Absorption of Sulphuric Acid by Wool—Results of Different Workers

is present, the amount of acid absorbed from solution increases and the pH of the equilibrating solution increases similarly. The desorption curve shows that a minimum absorption effect occurs below a liquor ratio of 20 : 1.

In Fig. 5, the absorption of acid by wool is plotted against the equilibrium concentration of the external solution. Up to acid strengths of ca. 0·1 M. these results agree with those of other workers. Above this concentration, however, the results indicate that at temperatures much above 0°C. some hydrolysis of the keratin may be taking place.

A comparison of the measure of acidity given by the pH of an aqueous extract and the acidity as determined by the pyridine method [15] is given in Fig. 6: both sets of results lie on smooth curves and can be used to correlate similar results, providing that certain precautions are observed [16]. Since this work was completed, another method, claimed to be more accurate than the pyridine method, has been introduced [17].

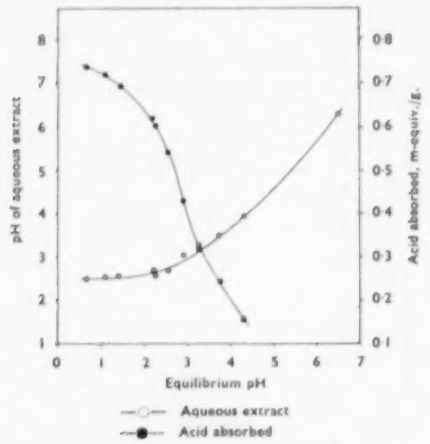

FIG. 6— Absorption of Sulphuric Acid by Wool— Comparison of the Measures of Acidity given by the Pyridine Method and by the pH of the Aqueous Extract

II. NEUTRALISATION OF ACID WOOL BY SOLUTIONS OF SODIUM CARBONATE

Large quantities (> 100 g.) of neutral wool were equilibrated for 24 hr. in sulphuric acid solutions of known concentration. The concentration of the equilibrating solution was determined, and the wool samples were then neutralised in solutions of sodium carbonate. The results are expressed graphically in Fig. 7–16.

(i) *Effect of Time of Equilibration* (Fig. 7–10)

Wools containing varying amounts of acid were equilibrated in solutions of varying strength. In all cases, apart from wool that was very acid (Fig. 10),

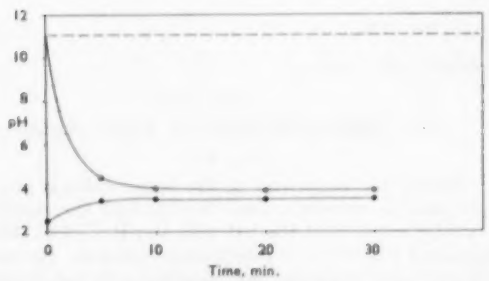

FIG. 7— Acid equilibration 0·05 M-H_2SO_4; alkali equilibration 0·01 M-Na_2CO_3

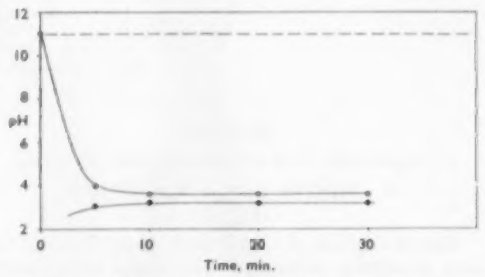

FIG. 8— Acid equilibration 0·25 M-H_2SO_4; alkali equilibration 0·01 M-Na_2CO_3

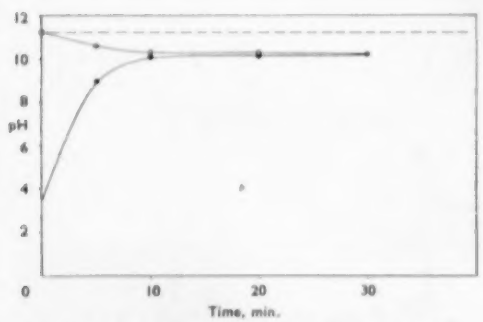

FIG. 9— Acid equilibration 0·01 M-H_2SO_4; alkali equilibration 0·1 M-Na_2CO_3

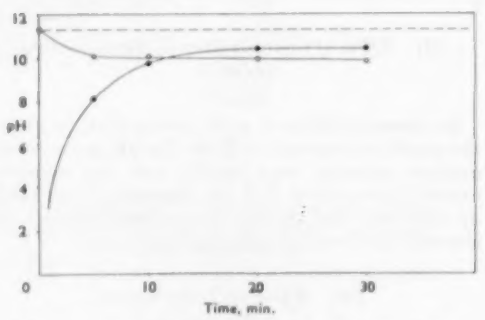

FIG. 10— Acid equilibration 0·25 M-H_2SO_4; alkali equilibration 0·1 M-Na_2CO_3

Original solution
Equilibrating solution
Aqueous extract

FIG. 7–10— Effect of Time of Equilibration on Neutralisation of Acid Wool by Sodium Carbonate (20°C., liquor ratio 20 : 1)

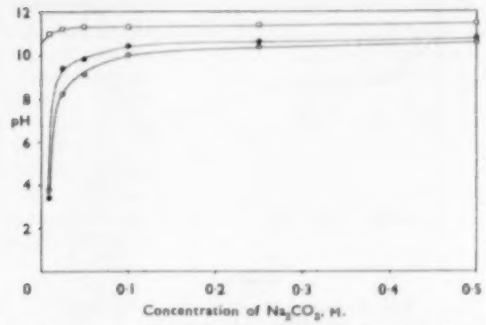

FIG. 11— Effect of Concentration (acid equilibration 0·1 M-H_2SO_4; 20°C.)

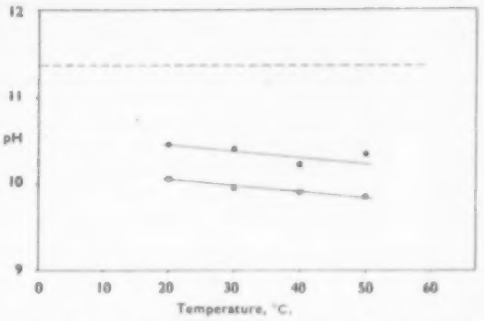

FIG. 12— Effect of Temperature (acid equilibration 0·05 M-H_2SO_4; alkali equilibration 0·1 M-Na_2CO_3)

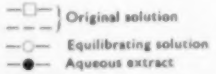

FIG. 11 and 12— Effects of Alkali Concentration and of Temperature on the Neutralisation of Acid Wool by Sodium Carbonate (liquor ratio 20 : 1; time of equilibration 30 min.)

equilibrium was reached in under 10 min. The final state of the wool, however, varied considerably. In most cases the pH of the aqueous extract showed that the wool was more acid (or less alkaline) than the equilibrating solution. In the case shown in Fig. 10, the pH of the aqueous extract is of exactly the same order as in Fig. 9, but the equilibrium pH is lower. This is due, as explained in a previous paper [11], to formation of bicarbonate (in this case due to acid desorbing from the wool) rather than to increased absorption of alkali. The actual amounts of carbonate and bicarbonate present at equilibrium can be calculated from the relations—

$$pH_1 - pH_2 = \log\left(\frac{[Salt]_1 - [OH]_1}{[Acid]_1 + [OH]_1}\right) - \log\left(\frac{[Salt]_2 - [OH]_2}{[Acid]_2 + [OH]_2}\right) \quad \text{(ii)}$$

and—

$$[Salt]_1 = [Salt]_2 + [Acid]_2 \quad \text{(iii)}$$

where the subscripts 1 and 2 refer to solutions before and after equilibration respectively.

Whether the pH of an aqueous extract is higher or lower than the equilibrium pH is a function of several factors [11], e.g. concentration of sodium carbonate, acidity of the wool, liquor ratio, and the carbonate–bicarbonate balance.

(ii) *Effect of Concentration of Equilibrating Solution* (Fig. 11)

Variation in concentration of the external solution, as shown by Fig. 11, gives rise, as would be expected, to a series of curves similar to those for an untreated wool. Only at a concentration of 0·01 M. sodium carbonate do the equilibration and desorption curves cross each other. In the particular case shown in Fig. 11, all higher concentrations give the type of curve found in Fig. 10. The results in Fig. 7 and 8 are systems covered by the extreme (low concentration) end of Fig. 11. Fig. 9 shows the case of a weakly acidic wool being neutralised by fairly strong alkali, where there is insufficient desorption of acid or absorption of alkali to reduce the equilibrium pH to a value below that of the pH of the aqueous extract.

(iii) *Effect of Temperature* (Fig. 12)

As described elsewhere [11], increase in temperature is accompanied by a small decrease in absorption of alkali. Although this is not the case for simple alkalis [10], the carbonate–bicarbonate buffering system has a negative $d(pH)/dT$ coefficient (and hence a lower effective hydroxyl concentration at higher temperatures), which appears to affect the normal increase in absorption with increase in temperature.

(iv) *Effect of Variation in Liquor Ratio* (Fig. 13–16)

With weakly acidic wools and strong solutions of alkali, the amount of alkali absorbed by the wool does not vary greatly, but the pH of the equilibrating solution falls rapidly with decrease in the amount of liquor present. The relative values of the measured pH again depend on the factors described under II (i) and (ii). With more strongly acidic wool (Fig. 14), the acid desorption from the wool becomes more important and the equilibration curve drops more steeply and to a lower value. With even more acid in the wool, initially, the effect is merely more pronounced, the results at a liquor ratio of 5 : 1 being very similar.

When the equilibrating solution is only weakly alkaline, the wool is never neutralised and the absorbed acid controls the whole system. The pH of the aqueous extract reflects this tendency, showing that there is insufficient alkali present to neutralise the acid involved.

DISCUSSION

Previous papers [4–7] have discussed fully the theoretical interpretation of absorption by wool from acid solution. Peters [7] and Olofsson [18–20] have put forward the relative merits of the Donnan and Gilbert–Rideal theories. In acid solution

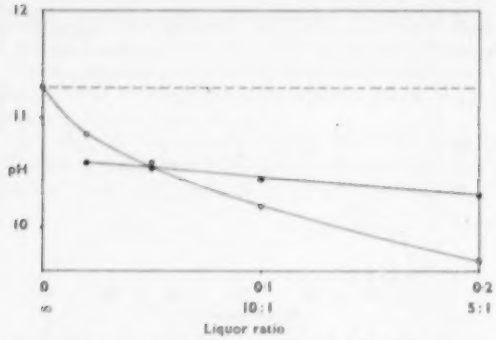

FIG. 13—Acid equilibration 0·0065 M-H_2SO_4; alkali equilibration 0·1 M-Na_2CO_3

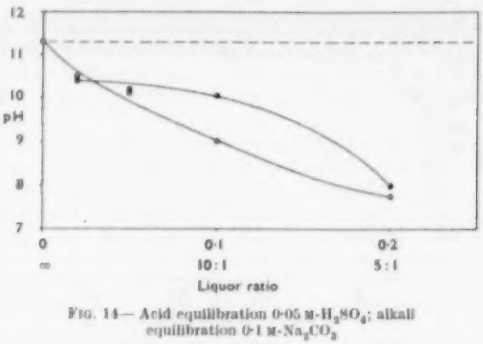

FIG. 14—Acid equilibration 0·05 M-H_2SO_4; alkali equilibration 0·1 M-Na_2CO_3

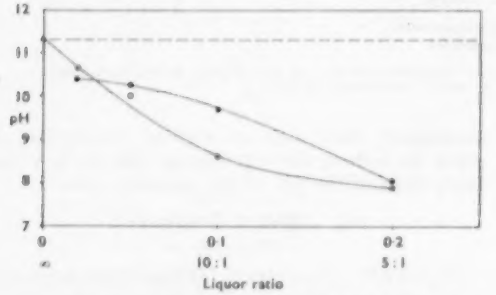

FIG. 15—Acid equilibration 0·25 M-H_2SO_4; alkali equilibration 0·1 M-Na_2CO_3

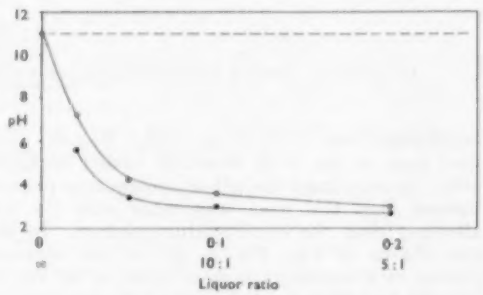

FIG. 16—Acid equilibration 0·05 M-H_2SO_4; alkali equilibration 0·01 M-Na_2CO_3

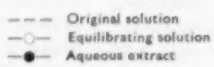

FIG. 13–16—Effect of Liquor Ratio on the Neutralisation of Acid Wool by Sodium Carbonate (20°C.; time of equilibration 30 min.)

(e.g. pH < 5) the Donnan theory gives a simple physical picture, which can be represented as follows—

CASE I

Internal Phase			*External Phase*
–COOH	$A\,\theta$		
$-COO^-$	$A\,(1-\theta)$	$[H^+] = x$	$[H^+] = X$
		$[SO_4^{2-}] = y$	$[SO_4^{2-}] = Y$
$-NH_3^+$		$[HSO_4^-] = z$	$[HSO_4^-] = Z$
Volume $= v$			Volume $= V$

The $[HSO_4^-]$ term can probably be omitted for pH values well above the pK value for $[HSO_4^-] \rightleftharpoons [H^+] + [SO_4^{2-}]$, i.e. pH > 2.

For isoelectric wool, equilibrated in sodium carbonate solution, the state at equilibrium can be represented as—

CASE II

Internal Phase			*External Phase*
$-COO^-$	A		
$-NH_3^+$	$A\,(1-\theta_1)$	$[Na^+] = c$	$[Na^+] = C$
$-NH_2$	$A\,\theta_1$	$[OH^-] = d$	$[OH^-] = D$
–OH	$[Ty]\,(1-\theta_2)$	$[HCO_3^-] = f$	$[HCO_3^-] = F$
$-O^-$	$[Ty]\,\theta_2$	$[CO_3^{2-}] = g$	$[CO_3^{2-}] = G$
Volume $= v$			Volume $= V$

In the case of acid-treated wool, brought to equilibrium in sodium carbonate solution (e.g. pH 9–11), the picture will be altered only by the addition of an $[SO_4^{2-}]$ concentration term, assuming that the acid is completely desorbed. The $[HCO_3^-]$ term will then include both the contribution due to absorption of alkali by the wool and also that due to neutralisation of CO_3^{2-} by H^+ due to desorption of acid.

For intermediate states (e.g. pH 4–9), where neither desorption of acid nor absorption of alkali can be complete, an intermediate diagram, with tyrosine [Ty] omitted (pK about 10·0), can be assumed—

CASE III

Internal Phase			*External Phase*
–COOH	$A\,\theta_3$		
$-COO^-$	$A\,(1-\theta_3)$		
		$[Na^+] = c$	$[Na^+] = C$
$-NH_2^+$	$A\,(1-\theta_4)$	$[OH^-] = d$	$[OH^-] = D$
$-NH_2$	$A\,\theta_4$	$[HCO_3^-] = f$	$[HCO_3^-] = F$
		$[CO_3^{2-}] = g$	$[CO_3^{2-}] = G$
		$[SO_4^{2-}] = h$	$[SO_4^{2-}] = H$
Volume $= v$			Volume $= V$

For all practical purposes, $[OH^-]$ can be neglected at pH < 8: below the neutral point, HCO_3^- will

begin releasing H^+ ($pK_1 \simeq 6{\cdot}35$) and the $[OH^-]$ term will be replaced by $[H^+]$. In all cases, θ represents the fraction of each of the groups which have combined. Concentrations in the external phase are expressed in gram-equivalents per litre and internal concentrations in milliequivalents per gram of dry wool: the volume v is assumed to be 0·285 millilitre per gram of dry wool [7].

If ψ is the potential difference between fibre and solution, the distribution coefficient λ between the electrically charged phases is [21]—

$$\lambda = \exp\left(\frac{\psi F}{RT}\right) \qquad \text{(iv)}$$

and in Case II, but including $[SO_4^{2-}]$ concentration terms, for an equilibrium at pH 10–11—

$$\lambda = \frac{C}{c} = \frac{f}{F} = \frac{d}{D} = \sqrt{\frac{g}{G}} = \sqrt{\frac{h}{H}} \qquad \text{(v)}$$

For electrical neutrality in the external solution—

$$F + 2G + 2H + D - C = 0 \qquad \text{(vi)}$$

If the parameter B (relating to the combining capacity of wool for base) is defined as such [7]—

$$\frac{B}{v} = (c-C) - (f-F) - 2(g-G) - 2(h-H) \qquad \text{(vii)}$$

$$= e - f - 2(g+h) - D \qquad \text{(viii)}$$

from (vi). "Internal concentrations" can be eliminated in equation (viii) by substituting the relations from equation (v), to give—

$$\frac{B}{v} + D = \frac{C}{\lambda} - F\lambda - 2(G+H)\lambda^2 \qquad \text{(ix)}$$

Application of the Donnan theory assumes differences in concentration between "internal" and "external" solutions, and the relation $\lambda = d/D$ can be written [7]—

$$\log \lambda = \mathrm{pOH_{ext}} - \mathrm{pOH_{int}} \qquad \text{(x)}$$

Knowing C, D, F, G, and H, equation (ix) can be solved graphically by plotting λ from 0·01 to 1·0 and obtaining the value of λ for a particular value of B/v: $\mathrm{pOH_{ext}}$ is known, and $\mathrm{pOH_{int}}$ can be calculated.

Equation (ix) assumes that all the acid formerly present has been completely desorbed. If acid is not present (i.e. $H = 0$), the equation reduces to the form—

$$\frac{B}{v} + D = \frac{c}{\lambda} - F\lambda - 2G\lambda^2 \qquad \text{(xi)}$$

which is the case when only sodium carbonate is present [11]. Also, if $F = G = 0$ and $c = D$, we have—

$$\mathrm{pOH_{int}} = 2\mathrm{pOH_{ext}} + \log\left(\frac{B}{v} + D\right) \qquad \text{(xii)}$$

which relates to salt-free absorption, or in the presence of salt [10]—

$$\mathrm{pOH_{int}} = \mathrm{pOH_{ext}} - \log C + \log\left(\frac{B}{v} + D\right) \qquad \text{(xiii)}$$

Application of these equations to the data relating to absorption from sodium carbonate solutions does not give very good agreement with Horner's data [10] for the saturation absorption of sodium hydroxide. With acid wools, the amount of base absorbed on neutralising with sodium carbonate was calculated from equation (ix), assuming that all the acid is desorbed, and gives rise to a corresponding amount of bicarbonate. These results, which are given in Fig. 17, agree with results obtained previously for absorption by neutral wool from solutions of sodium carbonate. The ($\mathrm{pOH_{ext}} - \mathrm{pOH_{int}}$) shift predicted by equation

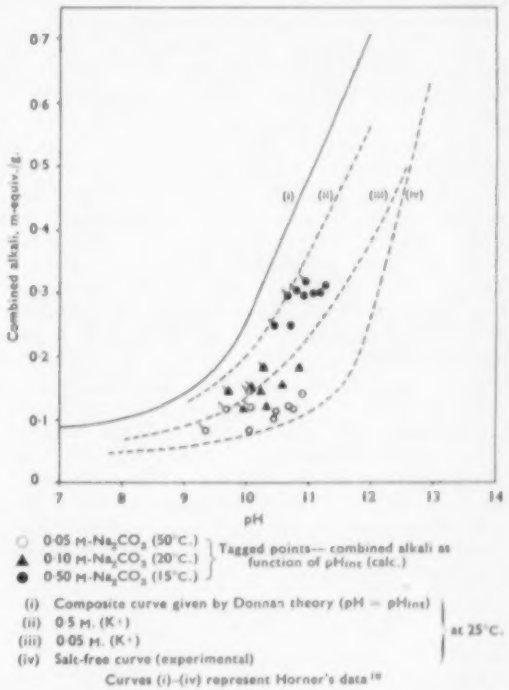

FIG. 17—Effect of Ionic Strength on Absorption of Alkali by Wool

(ix) is of the order to be expected, and the discrepancies mentioned above are very probably due to there being insufficient data available for activities of the various ion species under consideration. In the absence of reliable information for carbonate–bicarbonate systems, the mean activity coefficient for sodium hydroxide has been used as a measure of the hydroxyl concentration at a particular pH value. This is the most probable source of error, particularly when applied in equation (ii), where the term $[OH]_1$ in the denominator is of particular importance.

The assumptions that no CO_3^{2-} is absorbed and that sulphuric acid is completely desorbed are essential for the validity of equation (ix), and although this has not been demonstrated, the qualitative picture indicates that this is substantially true.

CONCLUSIONS

Information on the sulphuric acid–wool system is of particular interest to the dyeing [22] and carbonising [23–25] industries. The absorption of

acid by wool has received considerable attention, and more recently the uptake of acid by burrs has been studied [25]. The rate of absorption of acid by wool is fairly rapid, being mainly dependent on concentration, time, and liquor ratio. For a well opened wool, Pressley [24] suggests that it is substantially complete in about 4 min., but this figure may be slightly on the low side. Temperature would appear to have only a small effect.

The data relating to burrs show a more variable picture, but for burrs previously "wet-treated", the process may be a little faster than for wool [25]. Very little attention has been given to the neutralisation of acid-carbonised wool, and it is hoped that the foregoing results will be of practical use.

The concentration of neutralising agent (sodium carbonate) coupled with the time of immersion and the liquor ratio appear to be the rate-determining factors. In practice, the volume of the steep bowl will be fixed and the time for passage through the bowl will not, in general, be capable of wide variations. Temperature, again, does not seem to have any great effect. This leaves the concentration in the bowl and the weight of throughput as the only variables.

Over a long run, the pH of the neutralising bowl will fall slowly, owing to formation of sodium bicarbonate, but, provided that the salt concentration is maintained and the throughput is not too great, this should not affect the amount of alkali available for neutralisation, although the rate of absorption will probably decrease. The build-up of sodium sulphate concentration should not affect this process to any great extent [26].

Both the pH of an aqueous extract of the wool [12] or the pyridine method [15, 16] for residual acid can be used to give a measure of the state of the wool after a particular treatment. However, the problem of finishing the wool at any desired pH value is rather complex, and it must be emphasised that the pH of the neutralising bowl does not necessarily determine the final pH of the wool [11]. Such control can be achieved only by adjusting the alkaline salt concentration and/or by allowing sufficient time for desorption of acid and absorption of alkali to occur.

* * *

The author is grateful to Miss S. Braithwaite for experimental work and to Dr. F. O. Howitt for helpful comment.

Wool Industries Research Association
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ERRATA

Some Observations on the Absorption of Alkali by Wool Keratin from Aqueous Solutions of Sodium Carbonate and Bicarbonate, by R. P. Harker (J.S.D.C., **73**, 22 (Jan. 1957))

Equation (x) should read—

$$\log \lambda = pOH_{ext} - pOH_{int}$$

Line 32 should read—

$$\frac{C}{\lambda} - F\lambda - 2G\lambda^3 - D$$

In the key to Fig. 8 the open circles ○ should represent **0·05** M-Na_2CO_3 (50°C.)

Notes

Meetings of Council and Committees
November

Council— 6th
Perkin Main— 18th
Publications— 19th
Terms and Definitions— 1st
Dyers Company Research Medal— 6th

Notes for a Laboratory Course in Dyeing

Notes on laboratory experiments suitable for a course in practical dyeing, compiled by Dr. C. H. Giles, Senior Lecturer in Chemical Technology at the Royal College of Science and Technology, Glasgow, have now been published by the Society. Copies may be purchased at 17s. 6d. each.

Associateship Examinations 1958

Applications for the Associateship Examinations must be submitted by 28th February 1958. It is anticipated that the examinations will be held late in the month of May or early in June 1958. Intending candidates should communicate with the General Secretary for forms of application.

Perkin Centenary Awards 1958

The Perkin Centenary Trustees have announced the following awards for the academic year 1958–1959—

(1) Perkin Centenary Fellowship— One award is offered for one or two years to a graduate for the purpose of higher study. It has a value of not less than £600 per annum, and is tenable, from October 1958, at any university, technical college, or other institution, approved by the Trustees. Applications, on forms available from the Secretary, must be received not later than 1st May 1958.

(2) Perkin Centenary Scholarships— Two awards are offered, each for two years starting in October 1958 and renewable at the discretion of the Trustees for one further year, to enable candidates employed in an industrial firm or other institution concerned with the manufacture or the application of colouring matters to receive an education at a university or a technical college. Each award will have a value of £300 per annum. There is no means test for the award, and a successful candidate is not debarred from receiving the whole or a part of his normal salary from his employers during his tenure of the Scholarship. Applications, on forms available from the Secretary, must be received not later than 1st May 1958.

(3) Perkin Exchange Lectureships— An appointment will be made from time to time by the Trustees to enable a senior teacher at any appropriate university, or technical college, or other institution, to visit some similar establishment overseas in order to deliver a short course of lectures. In return, a visit to a British institution by some oversea scientist will be arranged. A second appointment contemplated will permit the exchange of lecturers for a period of up to one year between comparable institutions in Britain and overseas.

Inquiries relating to the above awards should be addressed to the Secretary, The Perkin Centenary Trust, c/o The Chemical Society, Burlington House, London W.1.

Annual Golf Competition

The Annual Golf Competition for the Goodall Trophy was held this year in conjunction with the Buxton Symposium on the course of the Cavendish Golf Club on Saturday, September 21st. Owing to a very heavy mist the start was greatly delayed, and this, in conjunction with the very wet condition of the course, resulted in a decision to play 13 holes only.

The Goodall Trophy was won by Mr. L. S. Newton (53 net). Second prize and best gross score were obtained by Dr. P. W. Smith (58 gross, 55 net). Third was Mr. G. E. Booth (56 net), and fourth Mr. R. Calvert (58 net).

Of the 34 entrants 24 actually played. The President sent his apologies for his inability to attend to present the prizes.

The meeting wished to record its appreciation of the kindness of Messrs. L. Morton Wood, R. K. Fourness, Fred Smith, and J. W. Reidy in presenting the extra prizes.

Worshipful Company of Dyers

At a General Court of the Company held on 9th October 1957, Lieut.-Col. A. R. Marshall, T.D., was elected Prime Warden and Lieut.-Col. H. F. Holme, M.A., T.D., Renter Warden.

U.S.A. Dye Imports

According to the Chemical Division of the Tariff Commission in Washington D.C. dye imports during 1956 declined in both volume (by 7%) and value (by 23%) compared with 1955. The total weight of dyes imported in 1956 was 2·8 million lb. (valued at $4·8 million), of which 49% was supplied by Western Germany, 42% by Switzerland, and 6% by the United Kingdom.

Ancient Peruvian Textiles

Textiles occupied an important place in the exhibition *Alt-Peru* held in the Zürich Art Gallery this Summer. Burial cloths from the cemetery of Paracas (A.D. 200–600) show outstanding colours, and within the gamut red–yellow–green–blue as many as 190 different colours have been distinguished. The Nazca culture developed almost parallel to this and is known especially for its paintings on textiles, which were taken from the highly developed ceramic art: this consisted largely of conventional demons, boldly drawn and faintly shaded with colour.

New Books and Publications

Safety in the Chemical Laboratory

By H. A. J. Pieters and J. W. Creyghton. 2nd edition 1957. Pp. xiv + 305. London: Butterworths Scientific Publications. Price, 40s. 0d.

Rather more than half of this book is devoted to a remarkably comprehensive review of the many factors which make for safety in the laboratory. Perhaps the most important of these is the cultivation by everyone of a sense of responsibility. The authors rightly emphasise that safety regulations are not in themselves sufficient—they must be practised at all times by all personnel. After dealing with general considerations of safety, they fully describe the precautions to be taken in using normal laboratory equipment and services, while the chapter on fire hazards is particularly well done. The discussion of explosive hazards goes well beyond, for example, the dangers of nitrations and of peroxides in ether, and its value is considerably enhanced by a list of potentially explosive combinations of chemicals. Safe procedures are outlined for handling a wide variety of corrosive, noxious, and toxic substances, and their physiological action is at the same time covered, even to the extent in many cases of describing their metabolism. The cumulative effect of many poisons and the development of sensitivity to particular substances are discussed, and several examples of the relationship between toxicity and chemical constitution are given. Protective equipment such as respirators, goggles, and dust masks is dealt with in detail. The general principles of first-aid start with the excellent advice that qualified medical attention should be sought as soon as possible; the symptoms and antidotes for specific poisons are tabulated. More detailed instructions for the disposal of waste materials would be valuable, as this is a recurrent problem in many laboratories. The methods of cleaning dirty apparatus might be advantageously enlarged upon, and the omission of reference to the traditional chromic acid "cleaning mixture" in the paragraph on the cleaning of glassware is curious.

There is much more in this book than the title would suggest. Some eighty pages are given to analytical procedures for toxic and hazardous substances, including the determination of toxic and other contaminants in air and in solution and the determination of dusts. The remainder of the book consists of tables which include summaries of safety considerations and the toxic concentrations and effects of a wide range of organic and inorganic compounds.

An extremely valuable feature of the book is the inclusion at the end of each chapter of a large number of references to sources of further information. In some respects, the index falls short of the standard set by the rest of the book, particularly as easy reference is implied in the italicised suggestion in the preface that the book should be used on the laboratory bench.

H. France

New Books received

Symposium on Industrial Water and Industrial Waste Water. ASTM Special Technical Publication No. 207. Philadelphia: American Society for Testing Materials. 1957. Pp. iv + 52. $2.00.

Textbook of Polymer Chemistry. F. W. Billmeyer Jr. New York: Interscience Publishers Inc. 1957. Pp. viii + 518. $10.50.

'Analar' Standards for Laboratory Chemicals. Poole and Chadwell Heath: British Drug Houses Ltd. and Hopkin and Williams Ltd. 5th edition 1957. Pp. xvi + 397. 21s. 0d.

Method of Test for Curl in Textile Fabrics. B.S.2888. London: British Standards Institution. 1957. Pp. 8. 3s. 0d.

Khimiya i tekhnologiya organicheskikh krasitelei. M. A. Chekalin. Moscow: Gosudarstvennoe Nauchno-tekhnicheskoe Izdatel'stvo Khimicheskoĭ Literatury. 1956. Pp. 576. 14 roubles 30 kopecks.

Laboratory Course in Dyeing. C. H. Giles. Bradford: Society of Dyers and Colourists. 1957. Pp. xi + 92. 17s. 6d.

Künstliche organische Farbstoffe. Band I. *Allgemeines Grundlegende Operationen, Wichtige Zwischenprodukte.* Band II. *Die wichtigsten Farbstoffe, ihre Herstellungen und hauptsächlichen Eigenschaften.* Zusammengestellt auf Grund der Vorlesungen von H. Hopff. Zürich: Verlag der Vereinigung der Chemiestudierenden an der Eidgenössischen Technischen Hochschule. 1956. Pp. x + 1–101 and viii + 105–319. 32 Swiss francs.

Wool through the Ages. London: International Wool Secretariat, Department of Education. 1957. Pp. iv + 80. 3s. 6d.

Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen. No. 381. *Zur Dichtebestimmung von Fasern. Methoden und Beispiele der praktischen Anwendung.* J. Juilfs. Opladen: Westdeutscher Verlag. 1957. Pp. 65. DM 17.00.

Technologie und Chemie der Synthetischen Fasern aus Polyamiden. H. Klare. Berlin: VEB Verlag Technik. 1954. Pp. 251 + 60 plates + 44 diagrams. DM 23.00.

Methoden der organischen Chemie. (Houben-Weyl). Band XI. *Teil* 1. *Stickstoffverbindungen II. Amine. 1 Herstellung.* Edited by E. Müller. Stuttgart: Georg Thieme Verlag. 4th edition 1957. Pp. lviii + 1178. DM 208.00.

Safety in the Chemical Laboratory. H. A. J. Pieters and J. W. Creyghton. London: Butterworths Scientific Publications. 2nd edition 1957. Pp. xiv + 305. 40s. 0d.

A History of the West of England Cloth Industry. K. G. Ponting. London: MacDonald & Co. (Publishers) Ltd. August 1957. Pp. 168. 15s. 0d.

Colour in Surface Coatings. Its Appreciation, Measurement, and Control. Teddington: Research Association of British Paint, Colour, and Varnish Manufacturers. 1956. Pp. 68. 7s. 6d.

Pigment Particles. Their Character and Behaviour in Paint. Teddington: Research Association of British Paint, Colour, and Varnish Manufacturers. 1956. Pp. 74. 7s. 6d.

Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen. No. 155. *Die auf Grau abgestimmte Farbwiedergabe im Dreifarbenbuchdruck.* K. H. Schirmer. Cologne: Westdeutscher Verlag. 1955. Pp. 33 + 3 plates. DM 10.00.

Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen. No. 193. *Untersuchungen über neue Pigmentfarbstoffe.* O. Schmitz-DuMont. Cologne: Westdeutscher Verlag. 1955. Pp. 37. DM 11.20.

Farbenlehre und Farbenmessung—eine kurze Einführung. W. Schultze. Berlin: Springer-Verlag. 1957. Pp. iv + 61. DM 9.80.

Organicheskie krasyashchie veshchestva. V. G. Shaposhnikov. Kiev: Gosudarstvennoe Izdatel'stvo teknicheskoĭ Literatury USSR. 4th edition 1955. Pp. 520.

Catalogue of Periodicals in the Shirley Institute. Manchester: Shirley Institute. December 1956. Pp. iv + 142.

Publications and Titles of Theses. 1954–5. University of Leeds. 1957. Pp. 73. 6d.

Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen. No. 161. *Über Silikone und ihre Anwendung in der Textilveredlung.* W. Weltzien and G. Hauschild. Cologne: Westdeutscher Verlag. 1955. Pp. 120. DM 27.00.

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Badische Anilin- und Soda-Fabrik AG.

Vialon Fast Dyes on Perlon Slubbing— This latest Vialon card (German text only) contains dyeings in three depths on Perlon staple fibre of 18 Vialon dyes together with 40 dyeings of combinations of them. In addition to the usual information, information is given for dyeing > 100°c., with a special note concerning the dyeing of Rilsan.

Vigoureux Printing on Wool Slubbing— This lavish card (German text only) describes the use of 10 Ortolan and 7 Basolan Chrome dyes for melange printing on wool slubbing. For each dye, samples of the finished blend are included to show the result obtained with 15% and 50% coverage, respectively. A solid dyeing to the same depth of colour as obtained on the printed portions is also shown for comparison.

Celliton Brilliant Yellow 8G— This disperse dye gives very bright, fluorescent greenish yellows on secondary cellulose acetate rayon, brighter and clearer than those obtainable using the 7G brand. In printing it is not dischargeable and is thus of interest for coloured discharges on dyed grounds as well as for direct printing. Fastness figures on secondary cellulose acetate include— Light (daylight) 4–5, washing b 3, sublimation 3–4.

Celliton Fast Red 4G— This disperse dye gives full reds on secondary cellulose acetate rayon, the wet fastness properties of the dyeings being superior to those obtainable with the older disperse dyes. Cellulosic fibres are slightly stained but this may easily be removed with hydrosulphite. On triacetate, dyeings are somewhat browner in hue and a dyeing temperature of 95–98°c. and a longer dyeing time is required for full depths. It is of interest for direct printing on secondary acetate and being an azo dye is dischargeable to white. Fastness figures on secondary acetate include— Light (daylight) 5–6, washing b 3.

Celliton Fast Grey GGU— This disperse dye gives greenish-greys on secondary cellulose acetate, the hue on triacetate being greener still. It is particularly level dyeing and the grey obtained is almost the same over a wide range of dyeing temperature thus reducing the risk of variations in hue arising from temperature differences between the selvedges and middle of the cloth. It is also of interest for direct printing on secondary acetate by the usual methods. Fastness figures on secondary acetate include— Light (daylight) 5–6, washing a 4–5.

Indanthren Brilliant Green 3GF— This vat dye gives bright greens on cellulosic fibres and is recommended for direct printing by the normal Rongalit C— potash method as well as by the Colloresin Process. It is also suitable for coloured discharges and coloured resists under Anthrasols and aniline black. Fastness figures on cotton include— Light 6, washing c 4–5, chlorine 3–4.

Helizarin Bordeaux R— This addition to the Helizarin pigment printing range gives bright bluish reds, the colour yield being high and the fastness to light and wet treatments of the prints obtained being of the same high order as with the other members of the series. Fastness figures for a print on cotton include— Light 6–7, chlorine 5.

Helizarin Red Violet R— This addition to the Helizarin pigment printing range is also suitable for pad-dyeing and is characterised by high colour yield and the excellent fastness of the prints and dyeings obtained. Fastness figures for a pad-dyeing on cotton and viscose rayon include— Light 7, chlorine 4.

Vialon Fast Blue FFG— This new member of the Vialon range of dyes for polyamide fibres gives very bright, pure blues having fastness characteristics of the same high order as those of the other members of the series with which it is fully compatible. It is also of interest for printing. Fastness figures include— Light 6, washing b 4, alkaline milling 4.

Ciba Ltd.

Deorlene Dyes— This card describes the properties and applications of this group of modified basic dyes to polyacrylonitrile fibres. Information is given on dyeing > 100°c. compound dyeings, the dyeing of mixtures of acrylic fibres and wool, cotton or viscose rayon, and polyamide fibres, and on stripping faulty dyeings. Five new dyes are included— Yellow G, Yellow 3R, Brilliant Red R, Blue BR and (in an appendix) Brilliant Red 4G. Deorlene Green JJO, which is described in the earlier card No. 2755/55, is omitted. The new members significantly widen the range, particularly in the yellow to red section.

Fastness figures for the new members include—

	Light	Washing	Milling
Deorlene		b	b
Yellow G	5	5	4–5
Yellow 3R	5	5	4–5
Brilliant Red 4G	4	5	4
Brilliant Red R	4–5	5	4
Blue BR	5	5	4–5

The Geigy Co. Ltd.

Maxilon Yellow 3GL; Red BL; Blue RL; Blue GL— The Maxilon dyes are a range of cationic (basic) dyes primarily designed for dyeing polyacrylonitrile fibres. The first four members described in this card build up well to give dyeings of any desired depth with good wet fastness characteristics. Except in the case of monofilament yarn the use of swelling agents or dyeing temperatures > 100°c. is unnecessary. Dyeing is carried out in presence of 2% of acetic acid (40%) and Tinegal NA or VR and Irgasol DA, the Tinegal NA or VR being increased and the Irgasol DA decreased with increasing amount of dye. They have good levelling properties, giving solid dyeings in combination. Since wool is well reserved they are particularly suitable for dyeing wool/acrylonitrile mixtures. Cellulosic fibres are also reserved. Fastness figures include—

	Light	Washing b	Permanent Pleating (Steam)
Maxilon			
Yellow 3GL	6	4–5	4–5
Red BL	7	5	4–5
Blue RL	7	5	5
Blue GL	7	5	5

Imperial Chemical Industries Ltd.

Brentamine Fast Red FR Base and Salt— This diazotisable base and the corresponding stabilised diazo salt are primarily of interest for producing scarlets and reds on cellulosic materials in combination with a wide range of coupling components. Selected combinations on cotton give dyeings of good to very good fastness to soda-boiling

and hypochlorite bleaching and also to light. They are of only limited interest in printing since in direct styles they offer no advantages over existing products and the Brenthols with which they exhibit outstanding fastness are unsuitable for printing owing to their high substantivity. Most combinations are not readily dischargeable.

Fastness figures on cotton include—

	Light	Chlorine	Soda Boiling
With Brenthol BC	4–5	4	4
With Brenthol BN	5	5	4–5

FD Caledon Powders Fine: Brands Currently Available, April 1957—Sixty-four members of this range are listed.

Caledon Rubine B—This vat dye gives bluish reds on cellulosic fibres. Dyeings have excellent fastness to light and to wet treatments and it is particularly recommended for "guaranteed" furnishings and casement materials. On cotton and linen it is best applied by Method 2 at 50°C. and on viscose rayon by Method 3 at 50°C. Above 70°C. progressively weaker and duller dyeings are obtained. The FD brand (the only form available) is suitable for pigment padding and pre-pigmentation application. It is not suitable for textile printing. Fastness figures on cotton include—Light 6–7, soda-boiling 4, hypochlorite 4–5.

Caledon Scarlet G—This vat dye gives bright scarlets on cellulosic fibres, maximum colour value being obtained by Method 2 or 3 at 20°C. Full colour yield is also obtained when vatting is carried out in full volume. It is particularly suitable for use in combination with Caledon Brilliant Orange 6R, Red BN and Red-Violet 2RN for jig-dyeing furnishings, casements, and dress goods and winch dyeing knitted materials. The FD brand is recommended for pigmentation methods. It is not recommended for dyeing materials subsequently to be soda-boiled. Fastness figures on viscose rayon include—Light 7, washing (Test No. 5, 5 times at 100°C.) 4–5 bluer.

QF Caledon Printing Scarlet G—This vat dye is a new member of the range specially prepared to give very rapid fixation when applied by the potassium carbonate–Formosul method and is supplied in a non-settling, non-drying form having a relatively high resistance to low temperature storage. It is of interest for direct printing, including pad-steam processes employing locust bean gum and similar products and as an illuminating colour in discharge styles. It is not suitable for dyed grounds subsequently to be discharged. Fastness figures on cotton include—Light 7, washing (Test No. 5, 5 times at 100°C.) 4 bluer.

Durazol Turquoise Blue GR—This direct dye gives brilliant blues of high fastness to light on cellulosic fibres. It is similar in dyeing behaviour (S.D.C. classification B) to Durazol Blue 8G but gives dyeings redder in hue and of somewhat better wet fastness, the fastness to water and washing being improved by an aftertreatment with Fixanol PN without any adverse effect on light fastness. It is also of interest on silk and nylon and for dyeing loose wool and slubbing to give bright greenish blues of high light fastness which cannot otherwise be obtained. It is also of interest for direct printing on cellulosic and protein fibres. Pale dyed grounds are dischargeable to white with neutral or acid Formosul. Fastness figures on viscose rayon include—Light 6–7, washing (Test No. 1, once at 40°C.) 3.

Durazol Green D—This direct dye (S.D.C. classification B) gives dull greens of good fastness to light on cellulosic fibres. It builds up well and is of particular interest for bottle-greens on viscose rayon staple cloths since subsequent crease-resist finishing causes only slight alteration in hue and the light fastness remains good at 5 in medium depths and the washing fastness is excellent. It is suitable for solid dyeings on cotton-viscose mixtures and is of some interest on unweighted silk. It is of little interest for direct printing. Dyed grounds cannot be discharged to white but coloured discharges are possible. Fastness figures on cotton include—Light 5, washing (Test No. 2, once at 55°C.) 3.

Lissamine Fast Scarlet R—This equalising acid dye gives bright yellowish scarlets of good fastness to light on wool yarn and cloth. On nylon also bright dyeings of good light and wet fastness are obtained but its building-up power is limited. Low solubility limits its interest for direct printing but dyed wool and nylon grounds may be discharged to white. Fastness figures on wool include—Light 6, washing (Test No. 1, once at 40°C.) 4, water 3.

Methasol Fast Blue 2B—A homogeneous spirit-soluble derivative of copper phthalocyanine giving greenish-blues of good light fastness in alcohols and glycols and in clear lacquers.

Monolite Fast Carmine BS Powder—This is a bright bluish red pigment of high tinctorial strength for use in paints and printing inks. It is also of interest for colouring linoleum, chalks, artists' colours and bookcloths. Light fastness figures include—Linseed oil (Madder 1–10 scale) 10, nitrocellulose (B.S.I. 1–8 scale), full shade 7–8.

Nylomine Dyestuffs—This range of anionic, directly-applicable dyes are specially designed for dyeing all forms of polyamide material to give bright colours of high fastness to light. Dyeings have good fastness to wet treatments which can be improved still further by a simple "back-tanning" treatment. The coverage of irregular-dyeing yarn is naturally not complete but is generally superior to that of existing anionic dyes. They are of limited interest on secondary cellulose acetate and, using the cuprous-ion technique, on polyacrylonitrile fibres. On nylon they provide a useful supplementary range for printing. The initial range consists of eight members, all of which are compatible in dyeing properties and exhibit similar fastness properties. Dyeing is carried out from neutral or faintly acid dyebaths according to depth, and "back-tanning" in a fresh bath containing formic acid and tannic acid to which tartar emetic is added after 30 min. treatment at 85–95°C. Fastness figures for a dyeing on nylon of a typical member of the range, Nylomine Red 2B, include—Light 6, washing (Test No. 2, once at 55°C.) 4–5, steam pleating (15 min. at 0·7 kg./cm.^{2}) 5.

Pure Primrose Chrome L6G—A lead chrome pigment possessing outstanding resistance to darkening on exposure inside and out.

Quinoline Yellow KT—The use of this acid dye for colouring paper in the beater is described. On sized paper it builds up well to give bright greenish yellows of good fastness and, unlike most acid dyes, does not require the use of dye-fixing agents to achieve this result.

Procion Red G—This reactive dye gives bright yellowish-reds of very good fastness to washing on cellulosic fibres. Dyeings on viscose rayon have good fastness to light but on cotton it is only moderate. It is capable of giving deep colours and may be applied by all the established methods, the best colour value in batch-wise application being obtained using trisodium phosphate. It is particularly recommended for dyeing silk on which deep dyeings are readily obtained. It may be applied to nylon but its use is limited. In printing on cellulosic fibres it is recommended as a standard red, light fastness being maintained in pale depths. Finished prints have good storage stability in acidic industrial atmospheres. Fastness figures include—For a dyeing on viscose rayon: Light 6, washing (Test No. 5, 5 times at 100°C.) 4–5. For a print on cotton: Light 5, washing (Test No. 5, 5 times at 100°C.) 4.

Procion Brilliant Red 5B—This reactive dye gives bright bluish reds on cellulosic fibres similar in fastness properties those obtained with the 2B brand but much bluer in hue. In addition it gives much deeper dyeings when dyed batch-wise. The presence of copper ions in the dyebath results in much weaker dyeings being obtained. It is of interest on silk and nylon but its use is limited by the light fastness being only moderate on silk and low on nylon. Fastness figures for a dyeing on cotton include—Light 4–5, washing (Test No. 5, 5 times at 100°C.) 4.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Nip Deformation of Rubber-covered Rolls. II—Investigations on the Press Section of the Paper Machine

B. Fahlin

Svensk Papperstidning, **60**, 460–466 (30 June 1957)

The measuring technique described in Part I has been used to enable the radial deformation of the rubber cover of a press roll to be calculated. The mathematical function describing the radial displacement of the pressure zone was found to be independent of machine speed and linear pressure between the rolls. An expression for the load distribution over the contact zone is derived. Variations of nip width and maximum normal pressure in the nip with linear pressure and machine speed have been investigated, and an equation is given for the water removal in the nip. G.S.B.

Automation in Bleaching and Finishing

V. G. Ivliev

Tekstil. prom., **17**, 30–35 (May 1957)

The design of a fully automatic bleaching and finishing plant for coarse calico, madapolam, and gauze is described in general terms. G.J.K.

PATENTS

Continuous Treatment of Viscose Yarns

Courtaulds *BP* 779,017

Apparatus is described for the liquid treatment of a continuous travelling yarn. W.G.C.

Wet Processing of Fibrous Materials

Kroy Unshrinkable Wools *USP* 2,742,773

The goods are carried through the liquor while being lightly held between two reticulated conveyors. During their passage through the liquor they pass between two pairs of rollers which are vibrated rapidly through a small amplitude. This gives quick, even treatment with good penetration. C.O.C.

Apparatus for the Wet Processing of Cloth and the like

Springs Cotton Mills *BP* 781,741

A machine in which the fabric enters at open width into and out of the liquor several times. The liquor flows in the opposite direction to the cloth and is channelled transversely widthwise of the cloth at each immersion. C.O.C.

Oscillating System for Impregnating Sheet Material

E. W. Smith *USP* 2,741,111

In a machine in which the cloth is led by guide rollers into and out of the liquor several times, treatment is much speeded up if the whole of the cloth in the machine is vibrated perpendicularly to its line of travel. A driving system for effecting this is described which is highly efficient and can be easily controlled. C.O.C.

Controlling the Operation of a Press Roll

Bowater Research & Development *BP* 783,421

Improved control of nip pressure in press roll operation is achieved by means of a movable link connected to a press roll carrier, the link being operated by a diaphragm which is deformable by controlled fluid pressure. The device also provides for movement of the press roll into and out of its operative position. G.S.B.

Heat Setting of Fabrics

Aetna-Standard Engineering Co. *BP* 782,003

A machine in which the distance over which the fabric is heated is automatically increased and decreased according to the speed of the fabric. Stretching is accomplished by means of the lead into and out of the oven. If the machine comes to a stop the fabric is immediately and automatically cooled and kept cool however long the machine is stopped. C.O.C.

Piece Dyeing Machine

Rodney Hunt Machine Co. *USP* 2,741,112

Two circular winches of the same size and the same height above the liquor co-operate with a separate specially designed plaiter to deliver the cloth at constant speed in folds to the back of the dye beck. This ensures that the fabric travels at constant speed and under constant tension from leaving the liquor until again entering it. C.O.C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Modern Detergents

F. C. Harwood

J. Roy. Soc. Arts, **105**, 407–481 (1957)

Historical, 17 references. C.O.C.

Chlorination of Phenol to Pentachlorophenol

N. Ohta and J. Imamura

Rept. Govt. Chem. Ind. Research Inst. Tokyo, **52**, 142–148 (xv) (April 1957)

A study of the synthesis of pentachlorophenol by the chlorination of phenol with chlorine gas is described. Although, even without the use of catalyst, phenol is easily chlorinated to trichlorophenol in a quantitative yield, in the absence of catalyst chlorination beyond trichlorination does not give tetra- or penta-chlorophenol, but tarry substances. In order that chlorination to pentachlorophenol may be successful, such a catalyst as $AlCl_3$, $FeCl_3$, $SbCl_5$, or Sb is necessary. C.J.W.H.

PATENTS

Size for Nylon

Monsanto *BP* 783,672

Terpolymers of vinyl acetate (50–52 mol. %), maleic anhydride (30–40) and a (β-alkoxy)ester of maleic acid (10–20) are good sizes for nylon yarn. They are readily removed during scouring. C.O.C.

Antistatic Finish

Boehme Fettchemie *BP* 779,491

A mixture of a polyalkylene oxide wax or a derivative thereof and a neutralised sulphonated fatty acid, or an ester thereof, is a good antistatic agent. It should contain $\leq$ 10 g./l. of the wax or its derivative. C.O.C.

BP 783,806

Fatty acids or alcohols of $>$ 10 C and containing at least two double bonds or their esters, ethers, amides, salts, etc., are useful antistatic agents, linoleyl alcohol giving excellent results on nylon. C.O.C.

Durable Antistatic Finish

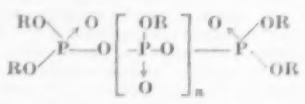
DuP *USP* 2,741,568

Polymers of ethylenically-unsaturated monomers of which at least 1% contain a vinylidene radical to provide the only non-aromatic C to C unsaturation, e.g. a poly-β-methacrylyloxethyldiethylmethyl ammonium salt, yield a durable antistatic finish on hydrophobic fibres. They are obtained e.g. by treating β-diethylaminoethyl methacrylate with dimethylsulphate and then polymerising. This product is then applied to the fibres which are then treated with a long chain carboxylic acid. C.O.C.

Antistatic Finish

DuP *USP* 2,742,379

Compounds of formula —



(n = 0–5; R = H or n-Alk of 8–18 C on the average half being H), e.g. the product obtained by treating a mixture of n-decanol, n-dodecanol, n-tetradecanol, hexadecanol and octadecanol with phosphoric anhydride, are good antistatic agents for use on non-cellulosic fibres. C.O.C.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

"Thermal" Rearrangement of Aromatic Hydrazo Compounds

H. J. Shine and R. L. Snell

Chem. and Ind., 706–707 (1 June 1957)

The statement of Vecera *et al.* (*ibid.*, 299 (1957)), that the action of heat on hydrazo cpd. both in the solid state and in soln. generally results in benzidine rearrangement, is critically discussed. Literature references (5) are cited to show that this is far from true, and work is described which indicates that in the cases of 1:1′(I)- and 2:2′(II)-hydroazonaphthalene, the benzidine rearrangement is not necessarily the prime reaction. I in ethanol gives mainly 1:2-7:8-dibenzocarbazole, a little 4:4′-diamino-1:1′-dinaphthyl, and a little 1:1′-diamino-2:2′-dinaphthyl, whereas II gives predominantly the 2:2′-diamino-1:1′-dinaphthyl in ethanol; in benzene the large yield of 65·5% reported by Krolik and Lukashevich (*Dokl. Akad. Nauk S.S.S.R.*, **65**, 37 (1949)) has not been confirmed. It appears that ammonia and carbazole formation are complementary reactions and probably take place where rearrangement occurs. H.H.H.

Dyes for Cellulose Acetate

K. Naiki

XI—Synthesis of Hydroxy-trichloroanthraquinones and their Amidation Products

Yûki Gôsei Kaguku Kyôkaishi, **14**, 34–9 (1956): *Chem. Abs.*, **51**, 7017–7019 (10 May 1957)

Dichlorophthalic anhydrides are condensed with *p*-chlorophenol in *sym*-tetrachloroethane with addition of $AlCl_3$. Ring closure in fuming H_2SO_4 gives hydroxy-trichloroanthraquinones but ring-closure under alkaline conditions gives dichloroxanthone-carboxylic acids. The OH group of the above anthraquinone compounds may be converted to OCH_3 using methyl-*p*-toluene sulphonate and one or two of the Cl groups may be replaced by NH_2 via the *p*-toluenesulphonamide derivatives. 1-Methoxy-4:8-diamino-7-chloro- and 1-methoxy-4:5-diamino-6-chloro-anthraquinones are red dyes for cellulose acetate. Reaction yields and product m.p. are given.

XII—Anthraquinonylcarbamic acid-2-chloroethyl ester

Ibid., 84–88

Cl·$COOC_2H_4Cl$ (chloroformic acid-2-chloroethyl ester) is prepared from ethylenechlorhydrin and phosgene. This reacts with 1-amino-2-methyl-, 1-amino-2-methoxy or 1-amino-2-bromoanthraquinone converting the 1-NH_2 group to –NH·COO·C_2H_4Cl. Heating with aq. KOH converts this group to –NHC_2H_4OH and the resulting products, e.g. 1-β-hydroxyethylamino-2-methylanthraquinone, are dyes for cellulose acetate. Affinities and fastness properties are compared, m.p. of products are given.

XIII

Ibid., 332–337

1:4-Diamino-2-methyl- and 1:4-diamino-2-methoxy-anthraquinone are reacted with Cl·$COOC_2H_4Cl$ and/or Cl·$COOC_2H_5$ to give 2-methyl- or 2-methoxy-anthraquinones having (in the 1- and 4-positions) -NH_2, -$NHCOOC_2H_4Cl$ or -$NHCOOC_2H_5$ groups. M.p. of all products are given. Affinities and fastness properties are compared. E.T.

Electrochemistry of Carotenoids

F. Körösy

Magyar Kémiai Folyóirat, **61**, 155–158 (1955): *Hungarian Tech. Abs.*, **8**, 6–7 (1956)

It was found that yellow-coloured carotene epoxides take on a bluish-green colour when dissolved in butyl alcohol containing sulphuric acid. The coloured substance obtained migrated in an electric field (50–90 v.) in the direction of the cathode. Other products formed during the oxidation of carotene behaved similarly. This cationic behaviour of the compounds was attributed to the formation of an oxonium salt on the ring oxygen atom. Carotenes and some of their oxidation products formed dark blue-coloured substances when dissolved in 85% sulphuric acid which migrated in the direction of the anode in an electric field. No satisfactory explanation could be given for this phenomenon. The dark blue-coloured sulphuric-acid solutions of the carotenes were extracted with benzene. Spectrophotometric investigation of the yellow-coloured compounds thus obtained showed, however, that they were not identical with the carotene compounds used as starting material. C.J.W.H.

Changes undergone by Chlorophyll (C.I. Natural Green 3) during Sterilisation in Tins

K. Heintze

Z. Lebensm.-Untersuch. u. -Forsch., **105**, 379–387 (1957)

A study of the splitting of Mg from chlorophyll (C.I. Natural Green 3), formation of phaeophytin colour during sterilising in tins, and effect of pH, Fe, Cu, Zn, Sn and Al ions. Spectral analyses showed that Fe and Sn replace the Mg of chlorophyll to yield brown and grey compounds respectively. These compounds can be differentiated from phaeophytin. Addition of $Cu(OH)_2$ to the infusion water during canning inhibits formation of phaeophytin and the influence of foreign metal ions on the colour of canned products containing chlorophyll. C.O.C.

Chrome Yellow and Chrome Orange Pigments

M. Kranz

Przemysl Chemiczny, (4), 174–183 (1955): *Polish Tech. Abs.*, No. 1 (21), 103 (1956)

Thirteen chrome yellow and chrome orange pigments such as lead chromate $PbCrO_4$, isomorphous compounds of the $PbCrO_4$,$PbSO_4$ type, and basic orange pigments of general formula $Pb(OH)_2$.$PbCrO_4$ were prepared. These were investigated and compared with $PbCrO_4$ and $PbSO_4$. All these compounds slightly catalyse the decomposition of hydrogen peroxide at 37°c. After activation of these pigments by adsorption on their surface of $[Fe(CN)_6]^{4-}$ and Cu^{2+} ions, multicomponent catalysts were formed which accelerated the decomposition of hydrogen peroxide. The method can be applied in technical control of individual chrome pigments. A characterisation is given of the disintegration of these pigments by physical methods (by determining specific gravity, apparent relative density, and sedimentation volume and by measuring speed of filtration). The light resistance of precipitates investigated was established after exposure to ultraviolet radiation for periods of 12, 40, and 100 hr., the changes being determined by the colorimetric method using a Lovibond–Schofield Tintometer and by the catalytic method through hydrogen peroxide decomposition. The sensitivity of the catalytic method is at least equal to that of the optical method. C.J.W.H.

PATENTS

Photographic Colour Developers

Gevaert *BP* 783,887

Compounds of formula Y·X·NH·CZ^1Z^2·COOM (M = H or water-soluble salt-forming cation; Z^1 and Z^2 = H, subst. or unsubst. Alk or Ar; X = subst. or unsubst. 1:4-arylene; Y = NR^1R^2 (R^1 and R^2 = Alk or together form a heterocyclic nucleus), e.g. the dihydrochloride of *NN*-diethyl-N^1-carboxymethyl-*p*-phenylenediamine, are excellent photographic colour developers. C.O.C.

Substituted *p*-Benzo-Quinones

FBy *BP* 783,042

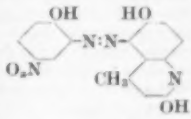
Phenol-*p*-sulphonic acids substituted in the *o*- and *o′*-positions by Hal, Alk or *cyclo*alkyl and which may also be substituted in the *m*- and/or *m′*-positions are oxidised to quinones with chromic acid or pyrolusite in acid solution. Oxidation takes place at 0 to 10°c. (or higher in certain cases) using the calculated amount of oxidising agent. Thus, 2:6-diethylphenol (200) are mixed with 100% H_2SO_4 (140) and heated at 100°c. for 30 min. The cooled melt is taken up in water (500) and the unreacted phenol separated by filtration. The filtrate is mixed with H_2SO_4 (220) and water (1000) and a solution of $Na_2Cr_2O_7$ (270) in water (500) is added dropwise at ≯ 10°c. After stirring 30 min. and standing overnight the 2:6-diethylbenzo-quinone is filtered off and purified by steam distillation. Yields and purities are good and the products are intermediates for dyes, medicines and photographic developers, fungicides and bactericides. E.T.

Metallisable Benzene-Azo-Quinoline Dyes for Wool, Nylon, etc.

FBy *BP* 783,753

2:6-Dihydroxy-4-methylquinoline, which may be made by cyclisation of *N*-acetoacetyl-*p*-aminophenol, is coupled with diazo compounds carrying in the *ortho* position to the diazo group a group capable of chelation, to give monoazo dyes which may be metallised with Cr or Co compounds

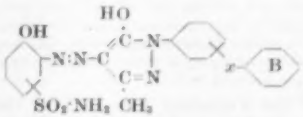
in substance, on the fibre or in the dyebath. The metal complexes of such dyes free from anionic water-solubilising groups dye wool and nylon from neutral or weakly acid baths. Thus, using diazotised 2-amino-4-nitrophenol the compound—



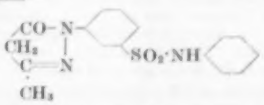
is produced, converted into its Cr complex by boiling its aq. NaOH soln. with aq. $K_2Cr_2O_7$ in presence of glucose. The product dyes wool black–brown. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.
Ciba *BP* 784,138

The Cr and Co complexes containing 1 atom metal to 2 mol. of monoazo dye, at least one of which is of the type—



(x = –NH·CO–, –SO_2–, –NH·SO_2–, or –O·SO_2–, the two latter groups being bound to the residue B by the S atom or by the N and O atoms respectively) dye wool and nylon yellow, orange and scarlet from neutral or weakly acid baths. Thus *m*-nitrobenzenesulphonyl chloride is condensed with aniline, followed by reduction of the nitro group, to give metanilanilide, which is diazotised and reduced to the hydrazine. Condensation with ethyl acetoacetate then gives 3-methyl-1-phenyl-5-pyrazolone-3′-sulphonanilide—



This is coupled with diazotised 2-aminophenol-4-sulphonamide, and the azo compound so formed boiled with aq. sodium chromosalicylate. Evaporation to dryness gives the soluble Cr complex which dyes wool orange. E.S.

Trinuclear Polymethin Dyes containing a Pyrryl, Inolyl or Pyrrocolyl Nucleus
Kodak *BP* 783,595

An additional way of making the dyes described in *BP* 756,226 (*J.S.D.C.*, **72**, 547 (1956)). C.O.C.

Hydrophobic Organophilic Pigments
American Cyanamid Co. *USP* 2,742,375

Products especially suitable for lithographic inks are obtained by slurrying pigments with an aqueous solution of a high mol. wt. pyridinium chloride, drying the treated pigment and then heating it for 5–30 min. at 100–200°C. C.O.C.

Crystalline Structure of Melanin, Keratin, and Collagen (XII p. 576)

V— PAINTS; ENAMELS; INKS

Stability of Pigment Suspension Aggregates in Low-molecular Organic Media
Ya. M. Gurevich

Colloid. J. U.S.S.R., **19**, 178–182 (March–April 1957)

It is suggested that the stability of aggregates of pigment suspensions in hydrocarbons, alcohols, and esters is determined by the adsorption of solvent molecules on the pigment surface. G.J.K.

Two-constant Theory of Kubelka and Munk and its Importance for Determining Coating Properties
F. Munk

Deut. Farben-Z., **11**, 84–90 (1957)

A mathematical formula is developed relating the amount of reflectance (Rx) of a coating with reflecting ability, light-scattering coefficient (S), amount of reflection of the base (R^1) and coating thickness (X). Rx plotted against X gave curves agreeing with experimental work. A formula developed for the case of a completely opaque film, is derived from the values of the two constants S and K (absorption constant). It is suggested that the theory can be the basis for determining the coating properties of white pigments. C.O.C.

Intumescent Fire-retarding Paint consisting of Protein

II— Relation of Fire-retarding Property of Casein to Degree of Hydrolysis and to Swelling in Dry Heat
N. Shikazono, H. Kamata, and T. Kanayama

Rept. Govt. Chem. Ind. Research Inst. Tokyo, **52**, 228–234 (xxv–xxvi) (July 1957)

The relation of the fire-retarding property of casein to its degree of hydrolysis and to the intumescent capacity of the hydrolysates was studied. The insoluble matter formed by the partial hydrolysis of casein with superheated water exhibited only a small capacity for fire-retardation and for swelling by dry heat, whereas the soluble matter simultaneously formed showed a large capacity for both. The fire-retarding ability of the soluble matter decreased as the hydrolysis proceeded, being closely related to the ratio of amino nitrogen to total nitrogen in the hydrolysate. In a restricted range, approximately a linear relation was observed between swelling volume and the fire-retardation time of test wood panels. A rapid and simple method is described for measuring the volume of a foam consisting of a carbonised casein skeleton.

III— Search for Substances which increase the Swelling Capacity of Casein Hydrolysate
N. Shikazono and T. Kanayama

Ibid., 235–241 (xxvi)

The swelling capacity in dry heat of casein partial hydrolysate, comprising the soluble and the insoluble matter, was increased by the addition of phosphoric acid, ammonium phosphate, some thermally unstable esters of phosphoric acid, and some organic bases. However, the other substances examined were quite ineffective in increasing the swelling: inorganic bases, salts, pigments, etc., all of which remain as ash after burning, hindered the swelling greatly; and inorganic acids other than phosphoric acid, organic acids, alcohols, aldehydes, etc. had generally little effect upon the swelling. When ammonium phosphate was used as swelling promotor, a film of the partial hydrolysate on a test wood panel exposed to flame not only swelled to form a carbonised foam of finer structure but also prolonged the fire-retardation time of the test wood panel. Thus, it will be possible to utilise casein hydrolysate without the removal of the insoluble matter. The effectiveness of phosphoric acid or ammonium phosphate in promoting the swelling was observed more markedly on the insoluble matter of the first hydrolysate of casein or on the soluble matter separated from the re-hydrolysate of the insoluble matter than on the soluble matter produced in the first hydrolysis. This suggests that the organic phosphorus originally contained in casein, although very small in amount, may be a factor affecting the swelling in dry heat of casein or its partial hydrolysate.

IV— Effect of Ammonium Phosphate upon Swelling in Dry Heat of many Proteins and their Hydrolysates
N. Shikazono, H. Kamata, T. Kanayama, and K. Maeda

Ibid., 242–246 (xxvii)

The swelling capacities in dry heat of many proteins and their partial hydrolysates containing added ammonium phosphate were measured. The swelling capacities of untreated proteins changed with increasing amount of ammonium phosphate added, in two different ways depending on the kind of protein. For some proteins, the swelling capacity decreased steadily with increasing amount of ammonium phosphate, whereas for the others it attained a maximum at a certain amount and thereafter decreased. Even in the case of casein, two modes of change of swelling capacity similar to the above were observed on many different preparations made by different manufacturers, but no apparent relation was found between swelling capacity and the constituents of the casein preparations. By the addition of the optimum

amount of ammonium phosphate, the swelling capacities of partially hydrolysed proteins were generally increased to various extents depending on the kind of protein and degree of hydrolysis. However, the swelling capacity of protein could not always be increased by partial hydrolysis. C.J.W.H.

Thixotropic Properties of Certain Printing Inks
A. A. Trapeznikov and T. G. Shalopalkina
Colloid. J. U.S.S.R., **19**, 232–243 (March–April 1957)

The strength and viscous properties of two printing inks are studied by means of an apparatus based on the coaxial cylinder principle. Stress–time curves are obtained showing the stress to pass through a value, corresponding to the shear strength P_r, to a quasi-equilibrium value P_s* exceeding a certain true value P_s. The structure of the ink can be gradually destroyed by higher velocity gradients, after which both P_r and P_s* increase in value during recovery. P_s* is shown to be capable of increasing also during continuous rotation, tending to attain the true equilibrium value. The thixotropic effect can be divided into—(*a*) the structural part, characterising a breakdown of the continuous network and evaluated by the difference between P_r and P_s*; and (*b*) the viscous part, reflecting the destruction and rebuilding of individual aggregates and evaluated by the difference in the P_s* values corresponding to a deformation of the completely (or partially) rebuilt, or completely (or partially) disrupted structure, and determining the total (or partial) width of the hysteresis loop. G.J.K.

PATENTS

Improving the Scrub Resistance of Poly(vinyl Acetate) Coating Compositions
Armstrong Cork Co. *USP* 2,739,910

Unexpected improvement in scrubbability (resistance to wet scrubbing with a brush) is obtained if the poly(vinyl acetate) is emulsified in an aqueous solution of sodium methyl siliconate. C.O.C.

Ruthenium for Colouring Glass Red and Black and for producing Brown Ceramic Stains
P. G. Herold, T. J. M. Planje, and J. C. Williams
USP 2,739,901

Addition of 0·001–20% of ruthenium or ruthenium oxide to lead borosilicate or lead silicate glasses, glazes and enamels yields pinks to reddish blacks. The blacks obtained have equivalent absorption of all wavelengths of light. Addition of 0·005–20·0% of ruthenium or its oxide to any one or a mixture of compounds of Si, Ti, Ge, Zr, Sn Ce, Hf and Th produces a brown ceramic stain. C.O.C.

VI—FIBRES; YARNS; FABRICS

Rigidity, Moisture, and Fibre Structure
R. Meredith
J. Textile Inst., **48**, T 163–T 174 (June 1957)

The relation between modulus of torsional rigidity and moisture regain for ramie, mercerised cotton, Fortisan, viscose rayon, wool, and nylon was investigated using torsion pendulums consisting of small bars suspended from the fibres in atmospheres of controlled humidity and temperature. The water absorbed is divided into two parts—*a*-water, which is associated with a definite group in the fibre molecule as in a chemical compound, so that its formation must be preceded by the rupture of a hydrogen bond in the fibre; and *b*-water, which fills spaces in the fibre under attractive forces like those in a liquid. For the cellulose fibres there is a straight-line relationship between the *a*-water absorbed and the relative rigidity of the fibre. For wool a further assumption is necessary: that the *a*-water is divided into *a'*-water, associated with the polar groups of amino-acid side-chains, and *a''*-water, associated with each amino-acid residue in the main chain. In the case of nylon, it is assumed that an appreciable fraction of the CO groups in the non-crystalline region cannot be hydrogen-bonded to neighbouring molecules, so that *a*-water is again divided into *a'*-water (associated with non-hydrogen-bonding CO groups) and *a''*-water (associated with hydrogen-bonding CO groups). Thus, as would be expected, where water replaces an interchain hydrogen bond in any of the six fibres, the torsional rigidity is lowered. Theories of water absorption are confirmed. P.T.S.

Effect of Temperature on the Initial Tensile Modulus of Some Wet Fibres
J. C. Guthrie
J. Textile Inst., **48**, T 193–T 200 (June 1957)

Initial tensile moduli (i.t.m.) of fibres have been measured in a water-bath over the temperature range 22–100°c. (Further experiments in a paraffin bath at 140°c. show the effect of removing the last traces of water from Orlon and Terylene.) Cellulosic fibres have i.t.m. which are greatly reduced by water, and temperature has only a secondary effect. Synthetic fibres are more sensitive to temperature and less to water. The breaking load of wet cellulose acetate is reduced, and its extensibility increased, by a rise in temperature. Compared with those in water, the i.t.m. and breaking loads of cellulose triacetate in dry-cleaning solvents are reduced. The i.t.m. of acrylic fibres are very sensitive to temperature and vary with the pH of the immersing solution. The i.t.m. of nylon is sensitive to water and temperature. The pH of the immersing solution has little effect on the i.t.m. of nylon and Terylene. The i.t.m. of regenerated protein fibres are affected by the pH of the immersing solution; they are reduced by increasing the temperature. The i.t.m. of the various fibres change with the length of time of immersion in water at a given temperature before the extensions are carried out. P.T.S.

Growth of the Cellulose Fibre. VI—Variation of Crystalline Region in Lint during Growth of the Cotton Fibre
Y. Ono
J. Soc. Textile Cellulose Ind. Japan, **13**, 127–129 (March 1957)

Photodegradation of Cotton Fabrics
I. Rusznák, M. Fehérvári, and F. Deák
Magyar Textiltechnika, (7), 255–257 (1955): *Hungarian Tech. Abs.*, **8**, 56 (1956)

Experiments were carried out to study the damage caused to cotton under the influence of light, air, and moisture on a bleached and on two dyed (one with Chloraminreinblau FF, the other with Chloraminrot 8B) cotton fabric samples. The degradation was evaluated by measuring the D.P. of the nitrated sample, by determining its copper number, and by measuring the carboxyl groups by the calcium acetate method. The decrease in the D.P. of the bleached cotton fabric was the most marked (from 2500 to 1150) after 40 hr. of illumination. The carboxyl content undergoes practically no changes during that time, whereas the copper count increases nearly seven–eightfold. Irradiation with ultraviolet in the presence of air is therefore really an oxidation process or at least is accompanied by such a process. Experiments carried out on dyed cotton fabrics yielded identical results. The rates of change of the D.P. and of the copper number are somewhat less for dyed cotton fabrics than for bleached fabrics, but the shape of the curves is similar. The rate of degradation was the lowest for the cotton fabric dyed red, for the blue fabric it was somewhat higher, and for the undyed it was the highest. On the basis of these observations it can be inferred that the degradation rate of cellulose during the irradiation may be checked to a certain extent by the oxygen consumption of the oxidisable dyes. C.J.W.H.

Acetylation of Cotton with Acetic Anhydride Vapour I—Acetylation with Sulphuric or Perchloric Acid Catalyst
I. Sakurada and Y. Sakaguchi
J. Soc. Textile Cellulose Ind. Japan, **13**, 13–16 (Jan. 1957)

Bleached cotton was immersed in an aqueous solution of sulphuric or perchloric acid, excess solution was pressed out, and the cotton air-dried to the desired degree and then brought into contact with acetic anhydride vapour at 30–60°c. for the acetylation. In contrast to the acetylation of rayon, various organic liquids such as lower alcohols and esters may be successfully employed instead of water for the pretreatment. Acetylation proceeds smoothly even when the water content of the fibre is as low as 10%. These observations indicate that liquids penetrate easier into cotton fibre than into rayon. But under such conditions that acetylation of both cotton and rayon proceeds smoothly, the rate of acetylation of the latter is greater.

II—Acetylation with Salt Catalyst and the Properties of the Acetylated Fibres
I. Sakurada and Y. Sakaguchi
Ibid., 67–72 (Feb. 1957)

Fibrous acetylation of cotton fibres with acetic anhydride vapour was carried out using $ZnSO_4$ and $ZnCl_2$ as catalyst. In the case of $ZnSO_4$ the acetylation proceeded smoothly, and highly acetylated fibres without serious damage were obtained, while in the case of $ZnCl_2$ it was difficult to attain any high degree of acetylation without using more concentrated catalyst solution. Reacetylation rate, chloroform and acetone solubility, and water absorbency of fibres which had been acetylated with various catalysts were compared. It seems that in the case of $ZnCl_2$ catalyst the amorphous part reacts selectively, so that the reaction of the crystalline part follows very slowly. Apparently, the type of reaction with sulphuric acid and $ZnSO_4$ is similar.

III—Acetylation of Cotton and Ramie Yarn
I. Sakurada, Y. Sakaguchi, and H. Yamaguchi
Ibid., 72–75

Fibrous acetylation of cotton and ramie yarn with acetic anhydride vapour was carried out using sulphuric acid and $ZnSO_4$ as catalyst. The count of yarn has scarcely any influence on the rate of the acetylation. Raw cotton yarn reacts somewhat slower than the bleached yarn. Evenness of dyeing of acetylated yarn is comparatively good. When $ZnSO_4$ was used, no loss of tensile strength was observed. The shrinkage of cotton yarn by acetylation increases with increasing degree of the acetylation and finally amounts to 20%, but this shrinkage may be avoided when some tension is applied during the acetylation. Ramie yarn can be acetylated as smoothly as cotton yarn, but the shrinkage is much less than in the latter.
C.J.W.H.

Liquid-phase Fibrous Acetylation of Cellulose
I—Acetylation of Viscose Rayon with Sulphuric Acid Catalyst
I. Sakurada and Y. Sakaguchi
J. Soc. Textile Cellulose Ind. Japan, 13, 194–199 (April 1957)

II—Acetylation of Viscose Rayon with Perchloric Acid Catalyst
I. Sakurada and Y. Sakaguchi
Ibid., 199–201

III—Fibrous Acetylation of Viscose Rayon after Displacement of Imbibition Water with Solvent Vapour
I. Sakurada, Y. Sakaguchi, and K. Nagai
Ibid., 292–297 (May 1957)

IV—Acetylation of Cotton Yarn with Sulphuric Acid as Catalyst
I. Sakurada and Y. Sakaguchi
Ibid., 297–301

V—Acetylation of Cotton Yarn with Perchloric Acid as Catalyst
I. Sakurada and Y. Sakaguchi
Ibid., 301–304

VI—Fibrous Acetylation of Cotton Yarn with Acetic Acid Diluent
I. Sakurada and Y. Sakaguchi
Ibid., 365–367 (June 1957)

VII—Properties of Acetylated Cotton Yarns
I. Sakurada and Y. Sakaguchi
Ibid., 367–370

Fibrous Acetylation of Viscose Rayon with Keten
III—Aspects of the Reaction
Y. Iwakura and I. Kanda
J. Soc. Textile Cellulose Ind. Japan, 13, 17–20 (Jan. 1957)

IV—Properties of the Acetylated Fibre
Y. Iwakura, M. Takikawa, and I. Kanda
Ibid., 20–23

C.S.I.R.O. Wool Textile Research
F. G. Lennox
J. Textile Inst., 48, P 312–P 320 (May 1957)

The research problems include work on scourable branding fluids; the contamination of wool by burrs; carbonisation; odourless fellmongering; scouring and its by-products (wool-wax, etc.); mechanism of water absorption by wool; mechanical properties of wool–creep, relaxation of stresses and crimp; the bilateral structure of the wool fibre; the crystalline : amorphous ratio in wool protein; the fractional extraction of wool proteins and the amino-acid distribution; studies of "model compounds", pure chemicals whose reactivity is compared with comparable structures combined in the wool protein; yarn manufacture using radioactively "tagged" fibres; wash-fastness of acid dyes; mélange printing; mothproofing fast to dry-cleaning; shrinkproofing by incorporating polymers in the fibres, by masking the scales to give the fibre a smooth surface, by giving the fibres uniformly rough surfaces, or by rounding the scale edges; substitutes for rabbit fur in felt hats; wool damage tests—the effect of temperature, pH value, and time, and methods for assessing damage; polarographic estimation of cystine and the estimation of amide groups and other methods of damage assessment. 66 References are given to the work described.
P.T.S.

Effect of Electrolytes on the Alkali Solubility of Wool
I. Shimizu and M. Oku
J. Soc. Textile Cellulose Ind. Japan, 13, 135–138 (March 1957)

The solubility of wool has been studied in 0·1 N-NaOH and 0·1 N-KOH at 65°C. each containing NaCl, KCl, KBr, KI, KNO_3, $NaNO_3$, K_2SO_4, or Na_2SO_4. The solvent power of electrolytes is relatively stronger at lower concentrations, and maximum solubility almost corresponds to the Hofmeister series. But when NaOH contains NaCl, Na_2SO_4, or $NaNO_3$, and KOH contains NaCl, KCl, or Na_2SO_4, the solubility of wool is less than in the absence of these electrolytes. When the ionic strength is below 1·8, the solubility of wool in alkali containing certain electrolytes may be satisfactorily described by the empirical equation—

$$\log S = \log S_0 + K_1\sqrt{\mu} - K_2\mu$$

where S is the solubility, S_0 the solubility in the absence of electrolytes, and K_1, K_2 are constants.
C.J.W.H.

Decomposition of Silk Fibroin by Sunlight. III—Mechanism of the Yellowing of Silk
S. Okamoto and S. Imai
J. Soc. Textile Cellulose Ind. Japan, 13, 139–143 (March 1957)

To explain the yellowing of silk, Chujo reported recently that tyrosine (13–14% contained in silk fibroin) was changed into the indole derivatives and then into melanin-like pigments photochemically. Tyrosine is easily decomposed in dilute hydrogen peroxide solution at room temperature, while tryptophan, especially its indole ring, is more stable. Silk cloth was therefore dipped in dilute H_2O_2 soln. to decompose tyrosine, and then exposed to ultraviolet radiation. The exposed cloth was sifted by means of the rate of yellowing and the reactions for the colouring matters such as nitric acid or Ehrlich's reagent. These changes were compared with those of oxidised wool as well as formalised silk or wool. Tyrosine and tryptophan fixed on rayon cloth by polyvinyl alcohol were also exposed to light, and any changes were detected by paper chromatography. It may be concluded that the main cause of the yellowing is not changes in the tyrosine, but rather tryptophan, although the amount of the latter contained in silk fibroin is very small.
C.J.W.H.

Swelling and Solution of Nylon
M. Kurokawa and T. Kawagishi
J. Soc. Textile Cellulose Ind. Japan, 12, 868–871 (Dec. 1956)

Nylon and vinylon fibres are placed on a glass plate. A few drops of an inorganic acid, e.g. H_2SO_4 of arbitrary concn., are dropped on them. On account of surface tension, the acid extends along the fibres, but after reaching some point which seems to depend upon the concentration of the acid, the temperature of reaction, and some other factors, the fibres begin to swell and dissolve from the further ends towards the original droplets. The mechanism still remains uncertain.
C.J.W.H.

Configurational Changes in Polyethylene Terephthalate
I. M. Ward
Chem. and Ind., 1102 (10 Aug. 1957)

It has been suggested that a *gauche* configuration of the—

$$-O-CH_2-CH_2-O-$$

group exists in amorphous polyethylene terephthalate in addition to the *trans*, which is the only configuration in crystalline material. Infrared spectral measurements on a cyclic trimer and on a linear oligomer are in agreement with these suggestions. W.R.M.

Demixing of Solutions of Mixtures of Polyvinyl Alcohol and its Derivatives
N. Mori and T. Tanaka
J. Soc. Textile Cellulose Ind. Japan, **13**, 130–134 (March 1957)

Demixing with such derivatives as polyvinyl formal, polyvinyl cyanoethyl ether, and partially saponified polyvinyl acetate was influenced by the kind of substituent, the degree of substitution, and the ratio of the two polymers. The properties of fibres spun from the solutions, such as the cross-sectional forms and the dyeability, were influenced greatly by the demixing in the fibre structure. C.J.W.H.

Acetalisation of Polyvinyl Alcohol
N. Nakamura

I—Acetalisation of Polyvinyl Alcohol Fibre with Benzaldehyde
J. Soc. Textile Cellulose Ind. Japan, **13**, 381–384 (June 1957)

Polyvinyl alcohol fibre was acetalised with benzaldehyde, using methanol–water as a solvent for the latter. Influences of the previous heat-treatment of the fibre and the composition and the temperature of the acetalisation bath were studied in detail. Emulsion of benzaldehyde was also used for the acetalisation. When the degree of acetalisation was higher than 30 mol. %, the fibres were resistant to boiling water.

II—Acetalisation of Polyvinyl Alcohol Fibre with *cyclo*Hexanone in Methanol Solution
Ibid., 384–387

Influence of the catalyst, solvent, and temperature on the fibrous acetalisation of polyvinyl alcohol with *cyclo*hexanone was studied. As *cyclo*hexanone is insoluble in water, mixtures of water and methanol were used as solvent. Degree of acetalisation was higher when methanol concentration and temperature of reaction were low, but it was difficult to obtain a higher degree of acetalisation than 30 mol. %. The resistance to boiling water of the fibres obtained was inadequate. C.J.W.H.

***Ftorlon*—a new Synthetic Fibre**
Z. A. Zazulina, R. N. Martsinkovskaya, and Z. A. Rogovin
Tekstil. prom., **17**, 6–7 (May 1957)

A fluorine-containing polymer of average mol.wt. 400,000–600,000 is prepared from a 12% solution in acetone by dry- or wet-spinning. Initial dry-stretching (300–350%) can be repeated (300–500%) at higher temp. (135–142°c.), increasing the tensile strength from 50 to 130 kg./sq.mm. Subsequent thermal relaxation in hot air (120–130°c.) for 1 hr. increases the extension at break from 10 to 17–20%. Ftorlon is extremely resistant to the action of HNO_3, H_3PO_4, H_2O_2, and NaOH, and is claimed to be superior to Chlorin in both chemical and thermal stability. Its resistance to light is higher than that of the polyacrylonitrile fibres; its moisture uptake is almost nil. G.J.K.

PATENTS

Protein Fibres of Improved Wet Strength
Courtaulds *BP* 782,456

The wet strength of regenerated protein fibres is improved by esterifying the protein and reacting the esterified protein with ammonia so that at least part of the esterified carboxyl groups are converted to amide groups. W.G.C.

Acrylonitrile Copolymers of Improved Dyeing Affinity
Montecatini *BP* 782,321

Polymers of acrylonitrile, vinyl pyridine and a third monomer, e.g. allyl or methallyl alcohol, have good affinity for disperse dyes. W.G.C.

Acrylonitrile Copolymers of Improved Dyeing Affinity
Carlisle Chemical Works *BP* 782,155

Fibres having improved affinity for basic, disperse and acid dyestuffs are obtained by spinning copolymers of acrylonitrile and a conjugated diene, e.g. 2-methylpentadiene-1:3, in which part of the double bonds of the copolymer have been converted to epoxy groups. W.G.C.

Action of Nitrous Acid on Groundnut Protein Fibre (X p. 573)

Crystalline Structure of Melanin, Keratin, and Collagen (XII p. 576)

Mechanism of Alkaline Degradation of Cystine Residues in Proteins (XII p. 576)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Effects of Phosphates and Silicates on Raw Silk. I—Effects of Condensed Phosphates on the Degumming of Raw Silk
A. Hirata and M. Kobayashi
J. Soc. Textile Cellulose Ind. Japan, **13**, 159–164 (March 1957)

The effects of condensed sodium phosphates, sodium metasilicate, and soap on the degumming of raw silk were investigated. As condensed salts, sodium pyrophosphate, sodium triphosphate, and sodium metaphosphate glass were used. The pH values, the permeating rate, and the sericin-dissolving power of phosphates, silicate, and soap were measured at the same concentration and temperature respectively. The results showed that the pH values of phosphates were lower than those of silicate and soap, the permeating rate of phosphates was greater than that of soap and smaller than that of silicate, and the dissolving power of silicate was the greatest and that of soap the smallest. Below 70°c. the solutions of phosphates and soap showed little or no dissolving power for sericin, but the solution of silicate dissolved sericin considerably. Treated with soap below 70°c., the weight of silk was increased by the contamination of free fatty acid or metallic soap. The factors which affect the dissolving power for sericin are considered to be pH values and permeating rate of the degumming reagents, but further investigations have to be made to make clear the mechanism of the reaction between phosphates and sericin. Risks are seen in either case (1) of dissolving a part of fibroin when treated with metasilicate solutions or (2) of contamination by fatty acid or metallic soap when treated with soap solution. The condensed phosphates, therefore, are considered to be better degumming reagents for silk. C.J.W.H.

Autoxidation of Hypochlorite Solutions
I. E. Flis and M. K. Bŷnyaeva
J. Appl. Chem. U.S.S.R., **30**, 339–345 (March 1957)

The autoxidation of hypochlorite at pH 6–8 and 10–50°c. takes place according to the equations—

$$2HClO + ClO^- \rightarrow ClO_3^- + 2H^+ + 2Cl^-$$

$$2ClO^- + HClO \rightarrow ClO_3^- + H^+ + 2Cl^-$$

The speed of the reaction is greatest at 10–35°c. T.Z.W.

Chlorine Dioxide in the Bleaching of Dissolving Pulps
H. Sihtola, Y. Hentola, N. Hakkarainen, N.-E. Virkola, A. Saarinen, G. Wigren, T. Ulmanen, and E. Saxén
Svensk Papperstidning, **60**, 412–419 (15 June 1957)

When the chlorine dioxide stage followed alkaline extraction in a multistage bleaching system, improved brightness of the pulp was obtained. A pH range of 5·5–6·0 and a temperature rise to 70°c. with a retention time of up to 3 hr. improved the bleaching, which caused only a negligible decrease in the α-cellulose content and viscosity of the pulp. Comparative tests with peroxide and hypochlorite bleaching, on the other hand, led to a marked decrease when bleaching was carried out to the same brightness level. G.S.B.

PATENTS

Bleaching
J. Dungler *BP* 781,716
The material is saturated with acid, superficially treated with a sodium chlorite or hypochlorite and then heated. This method effects considerable economy in the consumption of chlorite or hypochlorite. C.O.C.

Polyphosphates and an Alkali Salt of Ethylenediaminetetracetic Acid as Assistants in Peroxide Bleaching
Food Machinery & Chemical Corpn. *USP* 2,740,689
In the continuous peroxide bleach (steam bleach) uniformly high brightness is obtained with no objectionable chemical and/or mechanical damage to the fibre if the bleach liquor contains hydrogen peroxide, an alkali, a phosphate and a sequestering agent. C.O.C.

Dry Cleaning Cellulose Acetate with a Solvent containing a Gas Fume Fading Inhibitor
Celanese Corpn. of America *USP* 2,741,533
Addition of 2-amino-1-butanol to a dry-cleaning solvent gives good protection to the cleansed goods against gas-fume fading. Methyldiethanolamine and diethylethanolamine can also be used but do not give quite such good results. C.O.C.

VIII— DYEING

Measurement of Water Permeability of a Layer of Yarns coiled on a Cylinder
Y. Go, A. Shinohara, and F. Matsuhashi
J. Soc. Textile Cellulose Ind. Japan, **13**, 334–337 (May 1957)
In dyeing yarns coiled on a cylindrical bobbin or reel or in immersing in water raw silk or pot cake packed in the form of a cylinder before reeceling, water permeability is one of the most important problems. The flow through layers of cylindrical yarn may be expressed by the following equation, the gravity flow being neglected—

$$Q = \frac{2\pi klp}{\mu \log \gamma_e/\gamma_w}$$

where Q = total flux through the yarn layers in unit time, K = constant, l = width of layer, p = pressure head, μ = coefficient of viscosity, and γ_e and γ_w = outer and inner radii respectively. It has been found experimentally that this equation holds for sufficiently thick layers (> 3 rolls) at low Reynolds numbers. In case the thickness of the yarn layer is not uniform, for instance barrel or cone package, γ_e is a function of z (axis coincides with bobbin axis), and so when this function is known, we can obtain the Q–$\gamma_e(z)$ relation from the integration of $\gamma_e(z)$ along the z axis. C.J.W.H.

Diazotisation and Decomposition Reactions of Bases employed for Azoic Dyeing and the Continuous Diazotising Process
A. Pataki, I. Bozsó, and V. Feleki
Magyar Textiltechnika, (6), 214–217 (1955): *Hungarian Tech. Abs.*, **8**, 18 (1956)
The diazotising reaction of the bases forming soluble salts with hydrochloric acid is of the same order. Neither increases in the concentration of the nitrite or of the hydrochloric acid nor rises in the temperature alter the rate of the reaction in concentrations employed in practice. The rate constants of the diazotisation and decomposition reactions differ greatly; consequently it is possible to measure each step separately. In the continuous diazotising process the acidified solution of the base and the nitrite solution flow through a stirring device and react immediately. The diazotised solution of the base reacts within a minute or two in another stirring device with the buffer solution. The base prepared as mentioned above is immediately used for coupling. With the continuous diazotising method even those rapidly decomposing bases which could not be employed up to now can be used economically without any losses. C.J.W.H.

Dyeing Phenomena in Relation to the Crystalline Structure of Fibres. V— Dyeing Behaviour of the Crystalline Regions of Cellulose Fibres under Various Conditions
M. Mijasaka and M. Nakamura
J. Soc. Textile Cellulose Ind. Japan, **12**, 903–906 (Dec. 1956)
By using selected direct dyes the dyeing behaviour of the crystalline regions of natural and regenerated cellulose fibres gained by Maeda's method has been studied, with the following results— 1. The dye accessibility of a crystalline region varies with the dyeing conditions, the change being particularly marked when the salt content in the dyebath differs. The dye accessibility of a crystalline region is not always greater than that of a fibre itself. When dyeing is carried out under mild conditions, i.e. a low degree of exhaustion, the dye accessibility of a crystalline region is lower than that of a fibre itself, and *vice-versa*. 2. Addition of salt to the dyebath increases the absorption of direct dye on the crystalline regions of the fibre much more than it does on the fibre itself, and the covering effect on the surface is mainly observed, particularly in the crystalline regions. This effect is all the more evident as a dyeing is carried out at a higher temperature with the addition of more salt to the dyebath. These results seem mainly due to the change of the cohesive forces or that of the colloidal dispersed state of each dye molecule. 3. There is a wide difference in dyeing behaviour between the crystalline regions separated from a fibre and the crystalline regions contained in a fibre. This is presumably due to the nature of the crystalline region in the fibres, which is not fully in contact with dye molecules during dyeing, and the polar groups on the crystalline regions which probably remain inactive are not accessible to dye molecules. It seems hardly possible to ascertain the dyeing mechanisms in the micromolecular structure of fibres by these indirect dyeing experiments.
C.J.W.H.

Absorption Spectra of Dyes. III— Shifts in the Absorption Spectra of some Dyes in Cellophane on Dry Heating
Y. Ishii
J. Soc. Textile Cellulose Ind. Japan, **13**, 238–243 (April 1957)
Purified sheets of Cellophane are dyed with selected dyes. On dyeing, the wavelength of maximum absorption of the dyes is shifted to the red side, as compared with that in aqueous solution. The dyes are classified into three groups, according to the change in the absorption curves on dyeing and dry heating— (*a*) On dyeing, the new absorption, which ceased after heating, is developed to the red side of the maximum, and consequently a colour change is observed. (*b*) The same new absorption is shown, but the change in the curve cannot be observed on heating. (*c*) After dyeing no such deformation of curve is shown, except the shift of the maximum wavelength and the change of absorption coefficient. In the case of the dyes of group (*a*), Azo-orseillin and Benzo Azurine G (C.I. Direct Blue 8), which are substituted by OH groups at the *ortho* positions of both azo linkages, the colour change is greatly improved by treatment with $CuSO_4$ solution. The above new absorption reaches the peak of the visible region by this treatment without deformation by heating. The new absorption of the dye of group (*b*), Brilliant Purpurine 10B (C.I. Direct Red 61), which has no OH group, is not decreased by heating or not affected by Cu treatment. In the case of the group (*c*), Congo Red (C.I. Direct Red 28), Benzo Purpurine 4B (C.I. Direct Red 2), Congo Corinth G (C.I. Direct Red 10), Congo Corinth B (C.I. Direct Violet 21), and Azo Violet (C.I. Direct Violet 32), containing some dyes that are substituted by an OH group *ortho* to the azo linkage, two new absorptions are observed on dyeing, heating, or Cu treatment.
C.J.W.H.

Effects of Fixing Agents on the Fading of Dyed Viscose Films
Y. Ishii
J. Soc. Textile Cellulose Ind. Japan, **13**, 110–115 (Feb. 1957)
The viscose films are dyed with selected dyes, such as Chrysamine G (C.I. Direct Yellow 1), Direct Brown M (C.I. Direct Brown 2), Benzo Cuprol Blue FBL (C.I.

Direct Blue 158), Benzo Copper Blue 2B (C.I. Direct Blue 168), etc. The film is treated with aqueous solutions of the fixing agents, dicyandiamide–formaldehyde resin and Sapamine KW, or a cation-active levelling agent, Repellat. The absorption spectra of the films are measured, and the effects on the spectra of these treatments and of exposure to ultraviolet radiation are discussed— (*a*) As a result of the treatments with the agents the maximum wavelength of spectra of dyes are shifted to shorter wavelengths, and the fading of the dyed films is accelerated by these agents. (*b*) By the modified treatment, in which copper salt is added to the bath of the fixing agents, the fading of the films is greatly decreased, but has no relationship to the shift of the spectra. (*c*) A weak new absorption is developed on the dyed films with copper blue dyes on the red side of the maximum wavelength of the absorption curves. This new absorption is extinguished by fixing treatments, or exposure to ultraviolet radiation. The reason for this phenomenon appears to be that the hydrogen bonding between the dyes and the cellulose chains is broken, and the dyes are deposited in the film by the fixing treatment or exposure to ultraviolet radiation. C.J.W.H.

Effect of Dyeing on the Permanent Set of Wool Fibres

S. Okajima and K. Kikuchi

I— Effect of Acid Dyes

J. Soc. Textile Cellulose Ind. Japan, **12**, 899–902 (Dec. 1956)

Commercial Acid Anthracene Red 3BL (C.I. Acid Red 158) and Brilliant Alizarine Milling Blue G (C.I. Acid Blue 127) were used. After it was confirmed that the data took the normal distribution to the first approximation, these data were treated stochastically, and the following result was observed— dyeing had little effect on the set-ability of wool fibres, when the fibres were washed thoroughly after being dyed and set in water at pH 9·2.

II— Effects of Acid Mordant Dyes

Ibid., **13**, 170–174 (March 1957)

The effect of mordant dyeing upon the set-abilities of the wool fibres was examined; Neolan Brown GRM (Ciba), Chrome Brown RH (C.I. Mordant Brown 33), and Eriochrome Azurol B (C.I. Mordant Blue 1) were used as the dyes. In the case of Neolan Brown GRM, the set-abilities decreased a little as in the cases of the acid dyes (Part I). In this experiment the desorption of dyes was not observed during the setting. The effects of Chrome Brown RH and Eriochrome Azurol B were more marked than that of Neolan Brown GRM, but the effect appears to be negligible in the concentration ranges of dyes and mordants used commercially. As the mordant $Cr_2(SO_4)_3$ was also examined instead of $K_2Cr_2O_7$, but its effect is markedly inferior to that of the latter. C.J.W.H.

Experimental Data on Oxidation Dyes

I. Rusznák and E. Marton

Magyar Textiltechnika, (8), 285–287 (1955): *Hungarian Tech. Abs.*, **8**, 56 (1956)

The paper deals with the sources of fibre damage caused by oxidation dyes and with the possibilities of overcoming them. Salts formed by aniline with organic acids were prepared, and the nature of the oxidising agent, the temperature and time of oxidation, and the catalyst were varied. Fibre degradation was evaluated by breaking-strength tests. In the absence of mineral acids a black colour did not develop. After the experiments with aniline salts, tests were carried out with derivatives from which mineral acid is liberated during the oxidation, but only to the degree necessary for oxidation. The salts of aromatic amines with sulphamic acid were selected for this purpose; however, only in a pure state can they be used without causing fibre damages. The crude product contains impurities from which acid is liberated during the hydrolysis, thereby causing fibre degradation. During the course of the experiments with *p*-tolylsulphamic acid the authors also prepared the pigment itself and investigated whether this pigment could be used directly for printing. In respect of particle size the pigment was found to meet the requirements. The fastness of the printed samples was satisfactory. C.J.W.H.

Automotive Fabrics— Factors which affect Light-fastness

C. H. A. Schmitt

Amer. Dyestuff Rep., **46**, P 351–P 364 (20 May 1957)

An anecdotal review of the difficulties which the many synthetic-fibre types and the many possible cloth designs present to the dyer of fabrics for the car industry, with some remarks on light-fastness testing and colour matching. P.T.S.

Checking the Fading of Dyes— I

Z. Hajós and J. Fodor

Acta Chimica Academiae Scientiarum Hungaricae, **7**, 117–132 (1955): *Hungarian Tech. Abs.*, **8**, 40–41 (1956)

The retarding of the fading of dyes was investigated in aqueous solutions (distilled water was used) in the alkaline pH range by ultraviolet irradiation using a substantive dye as model substance. It was found that six Naphtol AS derivatives of the eleven examined exhibited a definite inhibitory effect. According to the experimental findings the inhibitory effect of the compounds can to a large extent promote the hydroxynaphthoic acid part of the molecule, but the anilide part of the molecule can also play a decisive rôle. In the latter case especially the group attached *ortho* to the acid amide grouping deserves attention. The extent of fading was reduced by autoxidation inhibitors, e.g. tricresyl phosphate, phosphoric acid, glucose, β-naphthol, thiourea, quinol, pyrocatechol, resorcinol, monobenzyl ether of quinol, etc. It was found that the compounds Katanol ON, Naphtol AS–OL, Indanthren Olive Green B, etc. ordinarily used for the retarding of fading simultaneously inhibit the autoxidation of benzaldehyde as well. Therefore it seems reasonable to assume that a certain correlation exists between the autoxidation processes and the fading of dyes; the inhibitory mechanism may be similar or identical. C.J.W.H.

Tendering of Dyed 6-Nylon on Exposure to Light. I— Tendering of 6-Nylon dyed with Disperse Dyes

M. Yoshida and T. Hashimoto

J. Soc. Textile Cellulose Ind. Japan, **13**, 165–169 (March 1957)

Aminoanthraquinone dyes increased the degree of tendering, while azo dyes decreased it. No relation was found between the light absorption of dyes and the degree of tendering. Correlation was found, however, between tendering and fading, when dyes were divided into two groups, aminoanthraquinone dyes and azo dyes. The photosensitivity of anthraquinone dyes appears to be mainly due to their anthraquinone nucleus. C.J.W.H.

PATENTS

Fixing Agent for Direct Dyeings

W. C. Durfee Co. *USP* 2,740,688

Modification of *USP* 2,090,396 which treats the dyeings with aluminium sulphate, an acid binder (e.g. Ca or Na acetate), a "shade balancer" (e.g. basic chromium sulphate), a tartrate (e.g. argol) and NaCl. In such compositions there is a tendency for the aluminium salt to hydrolyse and produce precipitates. This tendency is overcome by adding boric acid to the composition. C.O.C.

Improving the Wet and Light Fastnesses of Dyeings and Prints

General Aniline *USP* 2,741,535

Treatment with an aqueous solution of a Cu compound, an aminocarboxylic acid, and an acidic condensate of HCHO, dicyandiamide and a readily-ionised NH_4 salt improves the fastness to light and washing particularly of direct azo dyes. C.O.C.

High-temperature Vat Dyeing

General Aniline *USP* 2,742,340

Thermally unstable vat dyes, e.g. the dianthraquinonedihydroazines, can be dyed satisfactorily at 150–220°F. by maintaining the reduction potential at a predetermined optimum value, in most cases about 930–1050 m.v. by gradual addition to the vat of appropriate reducing agents, stabilisers and alkali. A mixture of hydroxylamine and sodium nitrite is the best stabiliser for use in this process. C.O.C.

Photodegradation of Cotton Fabrics (VI p. 568)

Improvement of Nylon Textiles. IV—Effects of Heat Setting on the Dyeing of Nylon (X p. 574)

Reduced Consumption of Chrome Black in Leather Dyeing (XII p. 576)

IX—PRINTING

Vigoureux Printing
M. Alves Da Silva

Lanifício, **7**, 93–107 (Feb. 1957): *Tint. ind.* (28), 255–272 (June 1957)

A review covering machinery, pastes, designs, and steaming. An improved machine manufactured by the Société Alsacienne de Constructions Mécaniques (Mulhouse) is described. S.R.C.

Recording Characteristics of Colour Temperature on Colour Films
Y. Mizushima and N. Nakamura

Rept. Govt. Chem. Ind. Research Inst. Tokyo, **52**, 171–178 (xviii–xix) (May 1957)

PATENTS

Printing with Vat Dyes
Scott Bader & Co. *BP* 780,803

The fabric is printed with a paste containing a vat dye, thiourea dioxide (C.I. Reducing Agent 11), and a thickener. It is then dried, padded with caustic soda, steamed and oxidised. The absence of a reducing agent in the caustic soda bath enables azoic dyes to be printed with or alongside the vat dyes. C.O.C.

Carrier for Pigment Printing Pastes
Interchemical Corpn. *BP* 781,498

A pigment is dispersed in an aqueous solution of pH 5–11 and containing $\geqslant 2\%$ by weight of a water-soluble salt of an interpolymer of an olefinically-unsaturated carboxylic acid containing at least one activated carbon to carbon double bond and at least one carboxyl group and a polyalkenyl polyether of a polyhydric alcohol of > 3 C and $\geqslant 2$ hydroxyl groups. C.O.C.

Preparing Polyethylene for Printing
Celanese Corpn. of America *BP* 783,664

The polyethylene is treated with a flame at $\leqslant 4000°$F. under such conditions that the surface cools rapidly so that the material being treated is not distorted. The treated surface has good ink adhesion both immediately after the treatment and after prolonged ageing. C.O.C.

Colour Correction of Subtractively Coloured Photographic Images
Kodak *BP* 783,730

Planographic Dye-absorptive Matrixes for Imbibition Printing
Technicolor Motion Picture Corpn. *BP* 783,404

Experimental Data on Oxidation Dyes (VIII p. 572)

X—SIZING AND FINISHING

Size-film Tests
I. Kelen

Magyar Textiltechnika, (5), 166–170 (1955): *Hungarian Tech. Abs.*, **8**, 18 (1956)

A method has been elaborated for analysing the properties of the size independently of the yarn. Films were prepared from different sizes. The films were brought to the standard humidity, cut into 1-mm. strips, and subsequently tested on a fibre-rupture tester. The tensile strength of films formed from potato starch and maize starch is appreciably higher than that of films formed from flour. Furthermore, by increasing the solubility of the starch, the tensile strength of both potato and maize starch films at first increases, and after passing a maximum it decreases with increasing solubility. The addition of fat has a disadvantageous effect on the tensile strength of starch films. Besides the decrease of the tensile strength, the elongation also diminishes. C.J.W.H.

Rabbit Fibres. XIV—Relation between Thickness of Angora Rabbit Fibres and Carrotting
S. Ikeda, S. Okajima, and S. Kato

J. Soc. Textile Cellulose Ind. Japan, **12**, 895–898 (Dec. 1956)

Action of Nitrous Acid on Groundnut Protein Fibre
A. M. G. Kinnear

J. Textile Inst., **48**, T 201–T 202 (June 1957)

References are given to work tending to show that deamination of wool, silk and casein fibres by nitrous acid forms new cross-linkages between the protein chains in these fibres. The rate of extension of groundnut fibre at constant stress gives a measure of the "internal viscosity", and it is a convenient method of estimating the degree of cross-linking in the fibre. The extensibility of deaminated fibres is considerably reduced, and the effect is most noticeable at the extremes of pH value (4 and 8) investigated, where the extensibility of untreated, control, fibres is especially high. Deamination carried out in the presence of copper, chromium, or cobalt ions further increases the resistance to extension. P.T.S.

Introduction of Chelate Bond into Fibres I—pH Titration Curves of Silk Fibroin
N. Hojo

J. Soc. Textile Cellulose Ind. Japan, **13**, 102–106 (Feb. 1957)

pH titrations with KOH were made of fibroin, alkali-treated fibroin, and fibroin treated with urea–formaldehyde resin. During alkali treatment, new groups are produced. The active groups in resin-treated fibroin seem to be changed. From the slope of the titration curve, we may judge the degree of denaturation and the effect of resin finishing. The addition of metal ions lowers the pH titration curve of fibroin in the presence of KCl. The addition of metal ions to *o*-aminobenzoic acid, which has a free amino and carboxyl group, greatly lowers the lower-pH buffer region, and appreciable chelation occurs even in the strongly acid region. The pH titration curves of glycine anhydride show that no chelation occurs in the cases of Cu and of Al. Interaction of *o*-aminobenzoic acid with Cu and Al is greater than that of fibroin with the same metals.

II—Effect of Chelation on the Mechanical and Dyeing Properties of Silk Fibroin
N. Hojo and Y. Ozaki

Ibid., 106–109

Experiments were made to investigate the effect of the introduction of chelate bonds to silk fibroin on its mechanical and dyeing properties. pH titrations were made on silk fibroin with and without "heavy" metals in the presence of KCl. Then the strength and the elongation of the fibroin treated with "heavy" metals in solutions of various pH values were compared with those of the same fibroin in the corresponding pH solutions without metals. The strength of the treated fibroin increases in the acidic region in all cases in comparison with those of the fibroin untreated and treated without "heavy" metals. The elongation has a tendency to decrease. Chelation seems to be produced in the fibre, as the strength of the fibre increases in the pH region where we may expect chelation to occur. Chelation did not show bad effects on dyeing in the cases of Sunchromine Brown RH conc. (C.I. Mordant Brown 33) and of Alizarine Red S (C.I. Mordant Red 3). Cu, Al, and Fe were used as "heavy" metals throughout those experiments. C.J.W.H.

Cationic Softeners—their Secondary Effects on Textile Fabrics
AATCC Philadelphia Section

Amer. Dyestuff Rep., **46**, P 41–P 57 and P 64 (28 Jan. 1957)

Cationic agents are used in finishing as germicides, water-repellents, direct dye fixatives, and softeners. This paper describes the effect of cationic agents (quaternaries, amine, amide, and iminazoline derivatives) on such properties as the tear strength, the seam strength, the electrical resistance, the ageing properties, the resistance to damage by heat, and the effect on the fastness of dyes to ageing, heat, and light. Acrilan, Arnel, cotton, nylon, rayon, and wool fabrics were used. Tear strength is improved except with rayon (no change) and wool (slight

decrease), because of better stress distribution as a result of the lubricating effect of cationic agents. Cationic agents (except water-repellents) are effective antistatic agents, since they promote the formation of a film of condensed moisture on the fibre surface. Cationic softeners do not discolour fabrics, although some may on heating, depending on the agent and the fibre. There is no significant change in seam strength after application of cationic softeners. P.T.S.

New Developments in Textile Preservation

P. F. Klens and W. J. Stewart

Amer. Dyestuff Rep., **46**, 346–350 (20 May 1957)

Resistance to microbiological attack should be introduced without deleterious modification of the properties of a fabric—colour, smell, handle, wearing qualities, etc. Fungicides are tested by burial in soil or exposure to pure cultures which are known to attack textiles. Cellulose can be made resistant to microbiological attack by cyanoethylation or acetylation. Various copper compounds are good fungicides if the green colour is not objectionable. The solubilised copper complex of *N*-nitroso-*N*-phenylhydroxylamine, known as copper cupferron (Fungitrol 25), is not strongly coloured, it does not smell, it does not adversely affect wearing qualities, and it can be applied from solvent solution or water dispersion or in combination with water-repellent finishes. A new zinc fungicide is described—it is more potent than zinc naphthenate, and yet, like the latter, it is colourless—Fungitrol 50. The results of tests with it are tabulated. Quaternary ammonium naphthenate is an alternative for light-weight fabrics. Convenient fungicides can be applied with other finishes. For preservation from bacterial attack during textile processing much lower concentrations of fungicide are necessary. P.T.S.

Water-repellent Treatment of Textiles with Silicones — Studies on the Mechanisms of Two Processes

J. A. C. Watt

J. Textile Inst., **48**, T 175–T 192 (June 1957)

The hydrogen atom in polymethylsiloxane is reactive, e.g. with alkali—

$$-O-\left[\begin{array}{c}CH_3\\|\\Si-O\\|\\H\end{array}\right]_n- + H_2O \xrightarrow{\text{Alkali}} -O-\left[\begin{array}{c}CH_3\\|\\Si-O\\|\\OH\end{array}\right]_n- + H_2$$

and the elimination of water from two SiOH groups with the formation of a new Si–O–Si linkage causes gelling of the silicone. Other catalysts promoting gelation are the fatty-acid salts of lead, zinc, and tin. In the present work stannous naphthenate and octoate were used. Results indicate that the water-repellency is the result of a tube of gelled silicone around the fibres. Nylon, Terylene, cellulose acetate, cotton, and viscose rayon were used in the experiments. A second type of water-repellency caused by silicones is described. Polydimethylsiloxane—

$$-O-\left[\begin{array}{c}CH_3\\|\\Si-O\\|\\CH_3\end{array}\right]_n-$$

is without the reactive hydrogen atom of polymethylsiloxane, but with zirconium oxychloride as catalyst the silicone appears to be adsorbed on the surface of the fibre. Less silicone is required for effective water-repellency by this method. The molecules of silicone fluids are believed to exist at ordinary temperatures in the form of helices. As the temperature is raised, the helices elongate and are more easily adsorbed on the surface of the fibre. P.T.S.

Improvement of Nylon Textiles

IV—Effects of Heat Setting on the Dyeing of Nylon

T. Yasuda and Y. Sato

J. Soc. Textile Cellulose Ind. Japan, **13**, 338–344 (May 1957)

The amount of dyes absorbed at equilibrium, and the rate of dyeing, are decreased by dry-heat setting. It is suggested that changes in internal structure, especially crystallisation, are caused by heat setting; therefore the amounts of amino end-groups in the amorphous parts decrease, and then the diffusion paths decrease too. C.J.W.H.

V—Effect of Heat-setting on the Crease-resistance of Nylon Textiles

T. Yasuda

Ibid., 406–408 (June 1957)

Heat setting was carried out under several different tensions on nylon textiles. The crease-resistance of nylon textiles is improved by heat-setting. Comparing several textiles with each other which had the same residual shrinkage after the treatment, it was found that the crease-resistance increases with increased tension on the textiles while heated. C.J.W.H.

Finishing of Vinylons. I—The Effect of Resin Finishing of Vinylon by Swelling with Phenol

T. Ueda

J. Soc. Textile Cellulose Ind. Japan, **13**, 232–237 (April 1957)

Vinylon is swelled or dissolved by pyridine, nitrobenzene, phenol, cresol, etc., and phenol, etc. are used for increasing the penetration of dyes. The degree of swelling and the decrease of elongation and strength of vinylon is investigated by using phenol as a swelling agent. Blended fabric of equal parts of vinylon and spun viscose rayon, and viscose spun rayon fabric are used, and then vinylon is subjected to a conventional urea–formaldehyde resin process to observe the effect, which is found satisfactory, in accordance with a presumption that the small chinks in the skin-stratum of vinylon will be enlarged by this swelling agent and that the resin solution penetrating through the chinks will cause condensation in the interior of the sponge-stratum. The results may be summarised as follows—(*a*) In the case of swollen vinylon fabric, decrease in its strength occurs, but because of resin finishing the effect is more satisfactory than with unswollen vinylon. (*b*) Increase of the internal condensation of resin is greater than in unswollen vinylon. (*c*) The blended fabric is of more practical value than 100%-vinylon fabrics. (*d*) Initially condensed resin finishing shows better results than the uncondensed resin. C.J.W.H.

PATENTS

Use of Molten Metal Baths in Warp Sizing

Gebruder Sucker *BP* 779,649

The warps are treated with a molten wax or the like and then passed through a bath of molten metal to force the wax into the threads. C.O.C.

Rendering Cellulose-containing Materials Flame-resistant

Ciba *BP* 783,886

The process of *BP* 747,014 (J.S.D.C., **72**, 365 (1956)) is applicable to non-textile materials, e.g. paper. C.O.C.

Enhancing the Fastness to Washing of Flame-resistant Finishes

Ciba *BP* 782,418

Flame-resistant finishes produced with non-cyclic compounds of the urea group and phosphoric acids are given increased fastness to washing by aftertreating the impregnated material with a formaldehyde-triazine or formaldehyde-urea condensate or an ether of such condensate with a low molecular weight alcohol. To secure the best results each time the treated material is washed it should be so treated that the alkali metal ions are replaced by ions of a volatile base, e.g. by giving a final rinse with an ammonium salt. C.O.C.

Reducing the Felting Power of Wool

Holeproof *BP* 781,517

The wool is treated with dry chlorine until it has absorbed 0·2–1·5% by weight, scoured and dried. It is then impregnated with 1·0–3·5% by weight of a melamine-formaldehyde condensate and baked to cure the resin. C.O.C.

Reducing the Felting Power of Wool

O. B. A. Nilssen *BP* 782,028

The wool is treated with a dilute aqueous solution of a strong, high molecular weight, organic base, e.g. cetyldimethyl-ethylammonium hydroxide or cetylpyridinium hydroxide. Such bases diffuse only very slowly into the fibres and so the greater part of the action is on the surface of the fibres only. C.O.C.

Increasing the Felting Power of Wool
B. F. Goodrich Co. *BP* 779,208

Wet wool treated with 10% of a β-lactone has its felting properties much increased. Thus a batt of wool (100 parts) impregnated with water (168·5) and β-propiolactone (39·7) was steamed, hardened on one side for 2 min. and on the other for 1 min., milled with 4% H_2SO_4 for 36 min., neutralised with ammonia and rinsed, shrank 43·7% in length whereas a batt similarly treated in absence of β-propiolactone shrank only 25·5%. C.O.C.

Respiratory Pigments of Blood in Setting Permanent Waves
Turner Hall Corpn. *USP* 2,742,909

Hemoglobin and hemocyanin can replace the usual inorganic oxidising agents. Hemoglobin is active only at < pH 7 probably because it is only then that the iron-porphyrin radical becomes a hematin which is active as an oxidising catalyst. Hemocyanin, however, is active in both acid and alkaline conditions because the cupric form of a hemocyanin is reduced by the reduced keratin and changes in pH have little influence on the reversible reaction of the cupric to the cuprous state. C.O.C.

Durable Antistatic Finish (III p. 565)
Intumescent Fire-retarding Paint consisting of Protein (V p. 567)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Fractionation of High Polymers. I—Wood Cellulose
B. G. Rånby, O. W. Woltersdorf, and O. A. Battista
Svensk Papperstidning, **60**, 373–378 (31 May 1957)

Extraction of nitrated wood pulp by an alcoholic solution of ethyl acetate removes non-cellulosic and low-mol.wt. material and stabilises the nitrated pulp. Pre-extraction also results in improved fractionation. W.R.M.

Brown Stain on Wet Cellulose
H. R. Ambler and C. F. Finney
Nature, **179**, 1141 (1 June 1957)

The "brown stain" on wet cellulosic materials is due to the formation of a degradation product of cellulose at the wet–dry boundary and the capillary concentration of this or a similar product already in the material. W.R.M.

Structure and Decomposition of Cellulose
V. I. Ivanov
Izvestiya Akad. Nauk S.S.S.R. otdel. khim. nauk, 358–365 (March 1957)

Weak bonds, present in the macromolecular chains of cellulose to approx. 0·1%, which are due to the presence of (*a*) non-glucosidic links (ligneous cellulose) and (*b*) glucosidic links containing carboxyl and aldehyde groups in the positions 2:3:6, are held responsible for certain chemical transformations of cellulose in different media. Improved schemes for the oxidation of cellulose by N_2O_4 in acidic and alkaline media are claimed, and new applications of oxycellulose as ion-exchanger in medicine are suggested. 55 references are given. G.J.K.

Iodine Adsorption on Cellulose Fibres
S. Ishikawa

I—Iodine Adsorption on Pulps
J. Soc. Textile Cellulose Ind. Japan, **13**, 202–206 (April 1957)

II—Iodine Adsorption by Rayons
Ibid., 206–208

Intrinsic Viscosity of Cellulose Nitrate and its Relation to Degree of Nitration and Degree of Polymerisation
T. Ôkawa
J. Soc. Textile Cellulose Ind. Japan, **13**, 305–309 (May 1957)

Intrinsic viscosity of several kinds of cellulose nitrates in acetone was measured and related to degree of nitration and D.P. by an osmotic method. The following observations are made—(1) $[\eta] = KP^a$ over a D.P. range 90–3000 ($K = 1{\cdot}95 \times 10^{-3}$, $a = 0{\cdot}91$, N% = ca. 13·8%, 20°c., *c* in g./litre). (2) *K* is a constant independent of degree of nitration; *a* varies with degree of nitration (N%), and the following relations were obtained —(*a*) N% > 11, $a = 0{\cdot}835\ N^3 - 0{\cdot}162N + 1{\cdot}553$; (*b*) N% > 12, $a = 0{\cdot}93 - 0{\cdot}055\ (14{\cdot}14 - N)$; $K = 1{\cdot}95 \times 10^{-3}$. C.J.W.H.

Staudinger's Constant for Cellulose Nitrate
T. Ôkawa
J. Soc. Textile Cellulose Ind. Japan, **13**, 371–373 (June 1957)

The K_m values of cellulose nitrates in acetone are measured and related to their degree of nitration and D.P. The following relations hold for a nitrogen content of about 13·8% N, which is obtained usually with a nitration mixture containing HNO_3–H_3PO_4–P_2O_5—

(*a*) D.P. < 600, $[\eta] = 11 \times 10^{-4}$ P, (*c.* in g./litre)
(*b*) D.P. 600 ∿ 3000 $[\eta] = 0{\cdot}1 + 9{\cdot}2 \times 10^{-4}$ P

C.J.W.H.

Heats of Swelling and Solution of Cellulose Nitrate
D. I. Gal'perin and L. I. Mosev
Colloid J. U.S.S.R., **19**, 167–171 (March–April 1957)

Heats of swelling and solution of cellulose nitrate (containing 12% N) in dibutyl phthalate and acetone have been determined over the entire concn. range. One molecule of dibutyl phthalate blocks two O·NO_2 groups. In the formation of a monolayer, only O·NO_2 groups take part in the energy interactions with the given solvent. It is suggested that this type of interaction is not universal, but depends upon the nature of the active groups of the solvent and the degree of nitration of the cellulose nitrate. The temperature coefficients of the heat of solvation of cellulose nitrate in various solvents have been determined, and the results correlated with changes in the heat capacity of the system taking place on solution. G.J.K.

Cellulose Phosphate

I—Preparation of Cellulose Phosphate by the Urea–Phosphoric Acid Method
K. Katsuura and S. Nonaka
J. Soc. Textile Cellulose Ind. Japan, **13**, 24–28 (Jan. 1957)

The influence of the molar ratio of urea and phosphoric acid, the degree of compression, and the temperature and duration of preliminary drying on the phosphorylation of cellulose was studied. There was an optimum molar ratio for obtaining highly phosphorylated cellulose. When the molar ratio was constant, the sample prepared under conditions of higher temperature and shorter duration of reaction showed less degradation and higher reactivity. On the whole, the main reaction was very slow and by repeating the reaction three times a sample containing only 9·03% phosphorus could be obtained. All samples obtained were insoluble in water and dil. NaOH, and were fibrous. As a result of high-frequency titration, all phosphate radicals were known to be in the form of dibasic ester, and the existence of cross-linkage could not be recognised.

II—Thermal Stability of Cellulose Phosphate
K. Katsuura
Ibid., 28–30

The flameproofing phenomena of cellulose phosphate were analysed by the thermal balance and the results obtained were as follows—1. The curves showing weight loss against temperature for ammonium cellulose phosphate (1) were very different from those for cellulose linters (2). The decomposition of (1) began at a lower temperature than (2), but the continued decomposition curve of (1) rose more slowly than (2). It was almost impossible to prevent charring of cellulose fibre when it was directly exposed to flame, but the presence of phosphate radicals could prevent further burning after the fibre was withdrawn from the flame. It was recognised that the greater the degree of esterification and the higher the D.P., the greater was the thermal stability. 2. Higher mol.wt. are obtainable at higher temperature and with shorter duration of reaction. 3. The weight loss curves of ammonium and sodium salts of cellulose phosphates were very different from each other, the former effecting better flameproofing than the latter. C.J.W.H.

Biological Stability of Low-substituted Cellulose Ethers
G. A. Petropovlovskiĭ
J. Appl. Chem. U.S.S.R., **30**, 486–489 (March 1957)

Low-substituted cellulose ethers are sufficiently stable in the presence of the cellulose-destroying bacteria when the degree of substitution exceeds 3%. T.Z.W.

Properties of Low-substituted Methylcellulose
G. A. Petropavlovskiĭ and N. J. Nikitin
J. Appl. Chem. U.S.S.R., **29**, 1707–1716 (Nov. 1956)

Dilute (0·01–0·02%) soln. of low-substituted methylcellulose in NaOH are stable and are suitable for measurement of their viscosity. The mol.wt. of the methylcellulose calculated from the latter corresponds to that of the nitrates (in acetone) prepared from it. The mol.wt. of the methylcellulose dissolved in 6·5% NaOH soln. does not alter during freezing. This may be due to great elasticity of the system, which has a low D.P. (1200–1400). Conc. soln. (1–2%) of the methylcellulose in NaOH are not stable. When the action of atm. CO_2 is excluded, their viscosity diminishes asymptotically until it approaches a constant value; CO_2 in the atmosphere causes an increase in their viscosity and finally coagulation. Conc. soln. subjected to a low temp. gradually decompose owing to an oxidative destruction. The soln. of methylcellulose in 4·5% NaOH coagulates during heating; that in 6·5% does not. The films prepared from methylcellulose have good strength. Their water absorption increases with rise in the degree of substitution. T.Z.W.

Rendering Cellulose-containing Materials Flame-resistant (X p. 574)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Crystalline Structure of Melanin, Keratin, and Collagen
E. Meirowsky
J. Invest. Derm., **26**, 95 (1956): *Chem. Abs.*, **50**, 10824 (1956): *J. Soc. Leather Trades Chem.*, **41**, 333 (Sept. 1957)

Depilation of Sheep Skins with Acetic Acid
M. S. Carrie, J. B. Moore, and J. S. Chisholm
N.Z. J. Sci. Tech., **38B**, 19 (1956): *J. Soc. Leather Trades Chem.*, **41**, 218 (June 1957)

Reversible Configurational Changes in Poly-L-lysine Hydrochloride induced by Water
E. R. Blout and H. Lenormant
Nature, **179**, 960–963 (11 May 1957)

Poly-L-lysine hydrochloride films show definite changes in the positions and dichroism of the absorption bands characteristic of the peptide group when the water content of the films is altered. β-Polypeptide forms are believed to exist in the range 0–60% R.H., and measurements suggest that < 2 water mol. per amino-acid residue are absorbed. α-Helix forms occur when the films are exposed to 80–98% R.H., corresponding to 4–6 water mol. per amino-acid residue. If the water content is further increased, solution occurs.

A. Elliott, B. R. Malcolm, and W. E. Hanby
Ibid., 963

Results in general agreement with those of Blout and Lenormant, using heavy water. The rate of drying is, however, found to have a considerable effect. W.R.M.

Mechanism of Alkaline Degradation of Cystine Residues in Proteins
J. M. Swan
Nature, **179**, 965 (11 May 1957)

The amino acid $\alpha\alpha'$-dimethylcystine has been synthesised. If direct hydrolysis of the sulphur–sulphur bond or ionisation of a proton adjacent to a sulphur atom is the initial step leading to breakdown, then the amino acid should be as labile to alkali as cystine. If cystine degradation is initiated by ionisation of the amino-acid α-hydrogen atom followed by β-elimination of an unstable disulphide anion, the new amino acid should be much more stable to alkali than cystine, since the hydrogen atom essential for ionisation has been replaced by methyl. Such increased stability is observed. It is suggested that a β-elimination mechanism is important in the action of alkali on cystine and peptides and proteins containing cystine. Proteins such as wool might be stabilised to alkali if a reagent able to substitute cystine α hydrogen atoms could be found. W.R.M.

Reduced Consumption of Chrome Black in Leather Dyeing
J.C.
Przegląd Skórzany, (10), 225–227 (1954): *Polish Tech. Abs.*, No. 2 (18), 147–149 (1955)

Experiments conducted in Warsaw Polytechnic Colour Laboratory have established that sodium thiosulphate and soda ash added in suitable proportions to M-extra quality black (as used for dyeing chrome leathers) enhances the productivity of black and effects substantial savings in dyes without impairing the effects to be imparted to the colouring. Experiments on a semi-industrial scale on calf-skins, and on an industrial scale on box calf and other cattle-hide leathers, showed that the saving in black amounts to roughly 10%. C.J.W.H.

XIII— RUBBER; RESINS; PLASTICS

Structure of Linear Polymers
V. A. Kargin, A. I. Kitaĭgorodskiĭ, and G. L. Slonimskiĭ
Colloid J. U.S.S.R., **19**, 131–132 (March–April 1957)

Present-day views on the structural arrangement of polymer chains are criticised. G.J.K.

Configuration of Molecules and Formation of Structures in Solutions of Polyacrylates
V. A. Kargin and N. F. Bakeev
Colloid J. U.S.S.R., **19**, 133–137 (March–April 1957)

An electron-microscopic study of polyacrylic acid and of the sodium, barium, caesium, and quaternary ammonium polyacrylates. G.J.K.

Halogenoarylation of Unsaturated Compounds with Aromatic Diazo Compounds. II— Acrylonitrile in the Halogenoarylation Reaction
A. V. Dombrovskiĭ, A. P. Terent'ev, and A. M. Yurkevich
J. Gen. Chem. U.S.S.R., **26**, 3214–3218 (Nov. 1956)

The reaction of acrylonitrile with diazonium chlorides in acetone solution is found to be catalysed by $CuCl_2$ but not by the chlorides of Cu^{+}, Sn^{2+}, Co^{3+}, Mn^{2+}, Ni^{2+}, Cd^{2+}, Zn^{2+}, Al^{3+}. In addition to the expected aromatic halogeno compounds and phenols, α-halogeno-β-arylpropionitriles are obtained. G.J.K.

Polyvinyl Cyanoethyl Ether. IV— Effects of Alkali Pre-swelling of Polyvinyl Alcohol and of Addition of Surface-active Agents on the Vapour-phase Preparation of Polyvinyl Cyanoethyl Ether
M. Negishi, I. Sekiguchi, S. Yanagibori, and S. Okada
J. Soc. Textile Cellulose Ind. Japan, **13**, 310–317 (May 1957)

The effects of the pretreat-swelling of P.V.A. powder with NaOH and the addition of surface-active agents (Brij 30–35, Sanisol C, and Setamol W8) on the average value and the distribution of the degree of cyanoethylation in the vapour-phase preparation of the water-soluble polyvinyl cyanoethyl ether were studied. Although the pretreat-swelling and the addition of active agents have almost no effect on the average degree of reaction, the former increases strikingly the precipitation temperature of the water solution of polyvinyl cyanoethyl ether ($\sim$ 20 mol. %) in the swelling time of 0·5–2 hr., and the latter shows a small increase of the precipitation temperature. C.J.W.H.

High-molecular Compounds. XCIX—Polycondensation of Methylene Chloride with Benzene
G. S. Kolesnikov, V. V. Korshak, and T. V. Smirnova
Izvestiya Akad. Nauk SSSR otdel. khim. nauk, 375–382 (March 1957)

The polycondensation of methylene chloride with benzene in the presence of $AlCl_3$ leads to the formation of a chain polymer containing dihydroanthracene nuclei, the latter bringing the chain growth to a standstill and hindering the formation of branched and three-dimensional polymers. G.J.K.

XIV— ANALYSIS; TESTING; APPARATUS

Chrome Yellow and Chrome Orange Pigments (IV p. 566)
Intumescent Fire-retarding Paint consisting of Protein (V p. 567)
Size-film Tests (X p. 573)
Action of Nitrous Acid on Groundnut Protein Fibre (X p. 573)

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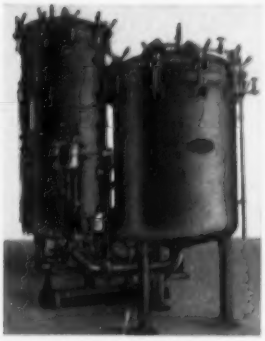

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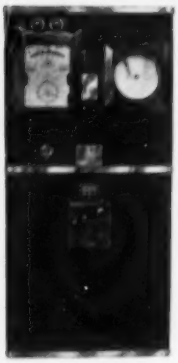

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FORTHCOMING MEETINGS OF THE SOCIETY— *continued from page* xii

Tuesday, 4th March 1958

LEEDS JUNIOR BRANCH. *Optical Bleaching Agents.* D. A. W. Adams, Esq., Ph.D. Department of Colour Chemistry and Dyeing, The University, **Leeds 2.** 3.15 p.m.

Thursday, 6th March 1958

SCOTTISH SECTION. *Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Scottish Woollen Technical College, **Galashiels.** 7.15 p.m.

Friday, 7th March 1958

BRADFORD JUNIOR BRANCH. *The Dyeing of Cellulosic Fibres with Alcian X Dyestuffs.* J. T. Turner, Esq. (I.C.I. Ltd.). Bradford Institute of Technology, **Bradford.** 7.15 p.m.

LONDON SECTION. *Recent Developments in the Dyeing of Man-made Fibres.* Dr. R. Wittwer (Ciba Ltd.). Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 11th March 1958

HUDDERSFIELD SECTION. *Recent Developments in the Dyeing of Man-made Fibres.* Lecturer to be announced later. Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

NORTHERN IRELAND SECTION. *Some Observations in the Uses of Synthetic Resin Products and Chemical Reactants to Cellulosic Materials.* F. Sloan, Esq., M.Sc. (Kirkpatrick Bros. Ltd.). Further details later.

Wednesday, 12th March 1958

SCOTTISH JUNIOR BRANCH. Annual General Meeting. 7 p.m.
Lecture 7.45 p.m. *The Drying of Textiles.* K. S. Laurie, Esq., M.A., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). Technical College, **Paisley.**

Thursday, 13th March 1958

MANCHESTER JUNIOR BRANCH. *Terylene.* J. R. Whinfield, Esq. Room C9, Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. Discussion, *Fluorescent Brightening Agents.*

Panel { A Member of the British Cotton Industries Research Association.
J. S. Ingham, Esq. (Marks & Spencer Ltd.).
Dr. Fearnley (Geigy Co. Ltd., Manchester).
J. Rayment, Esq. (G. & W. N. Hicking).

Victoria Hotel, **Bradford.** 7.30 p.m.

Tuesday, 18th March 1958

SCOTTISH SECTION. *Easy Care Finishes on Fabrics Containing Courpleta, with special reference to Pleating, Embossing and Setting.* A. S. Cluley, Esq., F.T.I. and S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). St. Enoch Hotel, **Glasgow.** 7.30 p.m. To be preceded by Sectional Annual General Meeting at 7 p.m.

Wednesday, 19th March 1958

MIDLANDS SECTION. *Optical Brightening Agents.* D. A. W. Adams, Esq., B.Sc., Ph.D. (Joint meeting with The British Association of Chemists.) Midland Hotel, **Derby.** 7 p.m.

Friday, 21st March 1958

MANCHESTER SECTION. One-day Symposium. *New Information on Finishing Processes.* Manchester College of Science and Technology, **Manchester.**

MIDLANDS SECTION. Section Annual Dinner. George Hotel, **Nottingham.**

Tuesday, 25th March 1958

HUDDERSFIELD SECTION. Annual General Meeting.

Thursday, 27th March 1958

BRADFORD JUNIOR BRANCH. *Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (I.C.I. Ltd.). Bradford Institute of Technology, **Bradford.** 7.15 p.m.

Thursday, 27th March 1958

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, **Bradford.** 7.30 p.m.

Saturday, 29th March 1958

BRADFORD JUNIOR BRANCH. Annual General Meeting. Bradford Institute of Technology, **Bradford.** 10.15 a.m.

Thursday, 3rd April 1958

MIDLANDS SECTION. *Recent Trends in Dyeing for the Carpet and Hosiery Trades.* W. Beal, Esq., B.Sc. (Geigy Co. Ltd.). Messrs. Carpet Trades Ltd. Canteen, Mill Street, **Kidderminster.** 7.30 p.m.
(Joint with the Textile Institute, Kidderminster Section.)

Wednesday, 16th April 1958

MIDLANDS SECTION. *Basic Principles of Dyeing with Disperse Dyes.* D. Finlayson, Esq., M.C., M.A., D.Sc. and H. C. Olpin, Esq., M.Sc., F.R.I.C., F.S.D.C. King's Head Hotel, **Loughborough.** 7 p.m. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section.)

Friday, 18th April 1958

LONDON SECTION. *Colour Fastness Requirements of Luton Textiles.* I. Glasman, Esq., A.T.I. (Marks & Spencer Ltd.). George Hotel, **Luton.** 7 p.m.

MANCHESTER SECTION. Annual General Meeting. Details to be announced later.

Friday, 2nd May 1958

LONDON SECTION. Annual General Meeting and Dinner. Aldwych Brasserie, Aldwych, **London, W.C.2.** 6 p.m. for 6.30 p.m. (Mr. C. O. Clark will speak on the standards required for the A.S.D.C. examination.)

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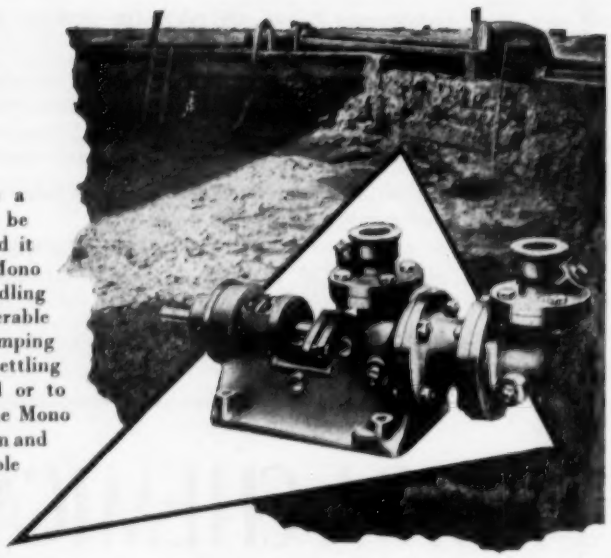

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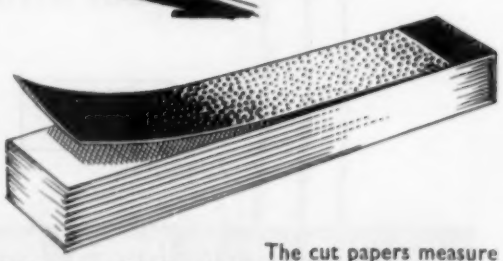

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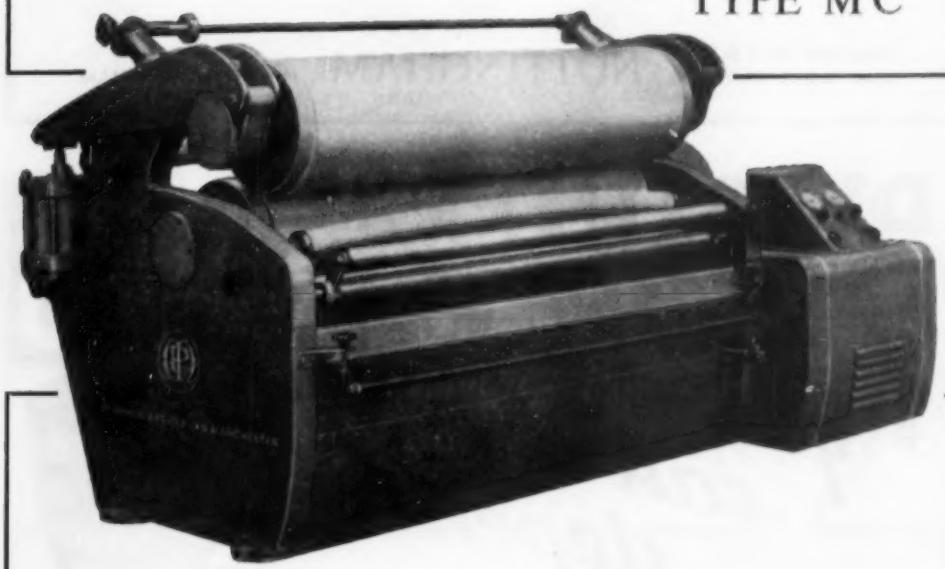

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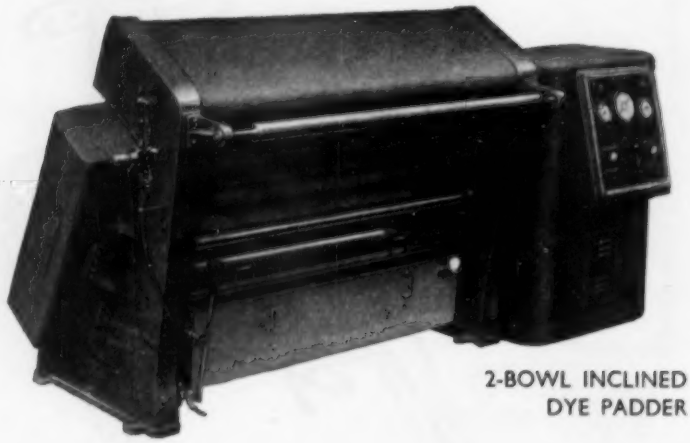

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